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IN UNCATALYZED CELLS(U) NAVAL SURFACE WEAPONS CENTER
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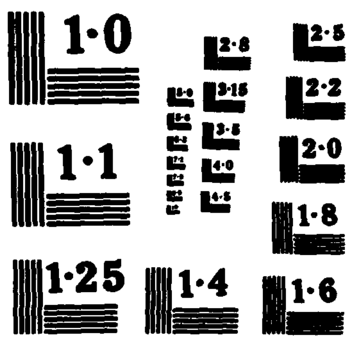

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NSWC TR 85-98

AD-A157 203

HIGH RATE $Li/SOCl_2$ CELLS
I. EFFECT OF DESIGN VARIABLES IN UNCATALYZED CELLS

BY W. P. KILROY (NSWC) L. PITTS K. M. ABRAHAM (EIC LABS, INC.)
RESEARCH AND TECHNOLOGY DEPARTMENT

APRIL 1985

NAVAL SURFACE WEAPONS CENTER
Dahlgren, Virginia 22448 • Silver Spring, Maryland 20910

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER NSWC TR 85-98	2. GOVT ACCESSION NO. AD-A157203	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) HIGH RATE Li/$SOCl_2$ CELLS I. EFFECT OF DESIGN VARIABLES IN UNCATALYZED CELLS		5. TYPE OF REPORT & PERIOD COVERED Nov 1983 - Apr 1985 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) WILLIAM P. KILROY (NSWC) AND L. PITTS AND K. M. ABRAHAM (EIC LABS, INC)		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Surface Weapons Center (Code R33) White Oak, Silver Spring, MD 20903-5000 and EIC Corporation, Norwood, MA 02062		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS		12. REPORT DATE April 1985 13. NUMBER OF PAGES 75
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution is unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Thionyl Chloride
Lithium Battery Safety

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Various cell parameters have been investigated to examine their effects on the performance, safety and chemistry of specially instrumented C-size Li/$SOCl_2$ cells. The temperatures, pressure and individual electrode voltages were monitored during discharge and forced overdischarge at ambient temperature and at -12°C.

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FOREWORD

This report examines the effect of design variables in high rate $Li/SOCl_2$ cells as part of the ongoing research program to determine causes of safety hazards in high energy lithium batteries.

Funding for this work was provided by the NAVSEA High Energy Batteries for Weapons Block Program.

Approved by:

J. R. Dixon

JACK R. DIXON, Head
Materials Division

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CHAPTER 1

INTRODUCTION

One of the goals of the Navy's High Energy Batteries for Weapons Block Program is to achieve a better understanding of safety hazards in high energy lithium batteries. To attain this objective, a statistical survey was planned to examine how various cell parameters (cell design, cell balance, catalysis, moisture content, etc.) would affect performance and safety in Li/$SOCl_2$ cells. Five replicate tests were planned for each cell parameter during various modes of testing including overdischarge, partial discharge and storage followed by further discharge, etc.

The statistical aspect of the research was curtailed due to a shortage of available funding.

Tests were performed using spiral and disk design commercial C-size $SOCl_2$ cells specially instrumented with a lithium reference electrode, a pressure transducer and internal and external thermocouples. For the purpose of comparing data, the surface areas of lithium and carbon and electrolyte volume were maintained the same in both the spiral and disk cells. This led to a poor disk cell design with suspected safety hazards. Consequently, tests on the disk cells were discontinued and most of the tests were carried out on the spiral cells.

Cell performance, and individual electrode potentials, cell internal and external temperatures, and pressure were measured during discharge and forced over-discharge at different rates at ambient and subambient temperatures. The effect of partial discharge and storage on cell performance was assessed. At various stages, the cell chemistry was compared using X-ray, gas chromatography, IR and mass spectroscopy techniques. The effects of water, the presence of a catalyst and lithium vs. cathode-limited cell balance were also examined.

The data have been summarized in two reports. This first report primarily addresses the effect of test conditions and cell parameters on performance and chemistry. The second report will discuss the effect of the catalyst.

CHAPTER 2

EXPERIMENTAL

CELL CONSTRUCTION

All cells were constructed by Hellesens Battery Engineering. A schematic diagram of the instrumented cells is shown in Figure 1. The C-size cells contained a vent mechanism, consisting of a 3 mil nickel membrane backed by a puncture spike, designed to vent between 275 and 300 psig. A stainless steel threaded bushing fitted with a removable plug was incorporated into the cell top. This allowed electrolyte sampling and the installation of a pressure transducer in a controlled atmosphere glove box. Miniature pressure transducers from Precise Sensors Inc., Monroe Ca., capable of measuring 0 to 500 psig were used to monitor the quick changes in pressure in such a small volume. The internal thermocouple was iron-constantan with a 304 stainless steel sheath of 0.010" diameter placed in a closed tube at the center of the cell (Figure 1). This 0.020" I.D. - 0.040" O.D. tube isolated the thermocouple from the electrolyte but it permitted rapid response to internal temperature changes. A small piece of lithium was attached to the outside of the tube to serve as a reference electrode. An additional thermocouple was placed outside the cell and held in place at the middle of the cell height by the heat shrinkable cell jacket. All other components of the cell construction and assembly had conventional characteristics of commercial cells.

The specifications of the cells are summarized in Table 1. The spiral wound cells had flat sheet lithium electrodes on a nickel grid placed between the separators and wound tightly into the cylindrical can. The carbon cathode was connected to the solid feedthrough while the lithium anode was connected to the cell can. The disk cells were essentially similar to the early Li - V_2O_5 cells.[1] The cathode disks, smaller in diameter than the internal diameter of the can, were fitted onto a central tubular post, while the anodes would fit tightly into the can. The round separator between each anode and cathode fit tightly against both the can wall and the central post. The component weights and surface areas of electrodes used in the disk cells were maintained as close as possible with those of the spirally wound cells. All the cells contained 1.8M $LiAlCl_4$ in $SOCl_2$.

The effect that water might have on the performance, safety or chemistry of $SOCl_2$ cells was examined by constructing "dry" and "wet" cells. The "dry" cells contained ~50 ppm water in contrast to the ~500 ppm water present in the "wet" cells. Dry electrolyte, containing less than 10 ppm of water, was used for all cells. The "wet" electrolyte was prepared from this dry electrolyte by carefully adding water in a controlled manner. The additional water content of the cell comes from water absorbed on the carbon and separator material. Proprietary drying techniques are reported to limit this water to approximately

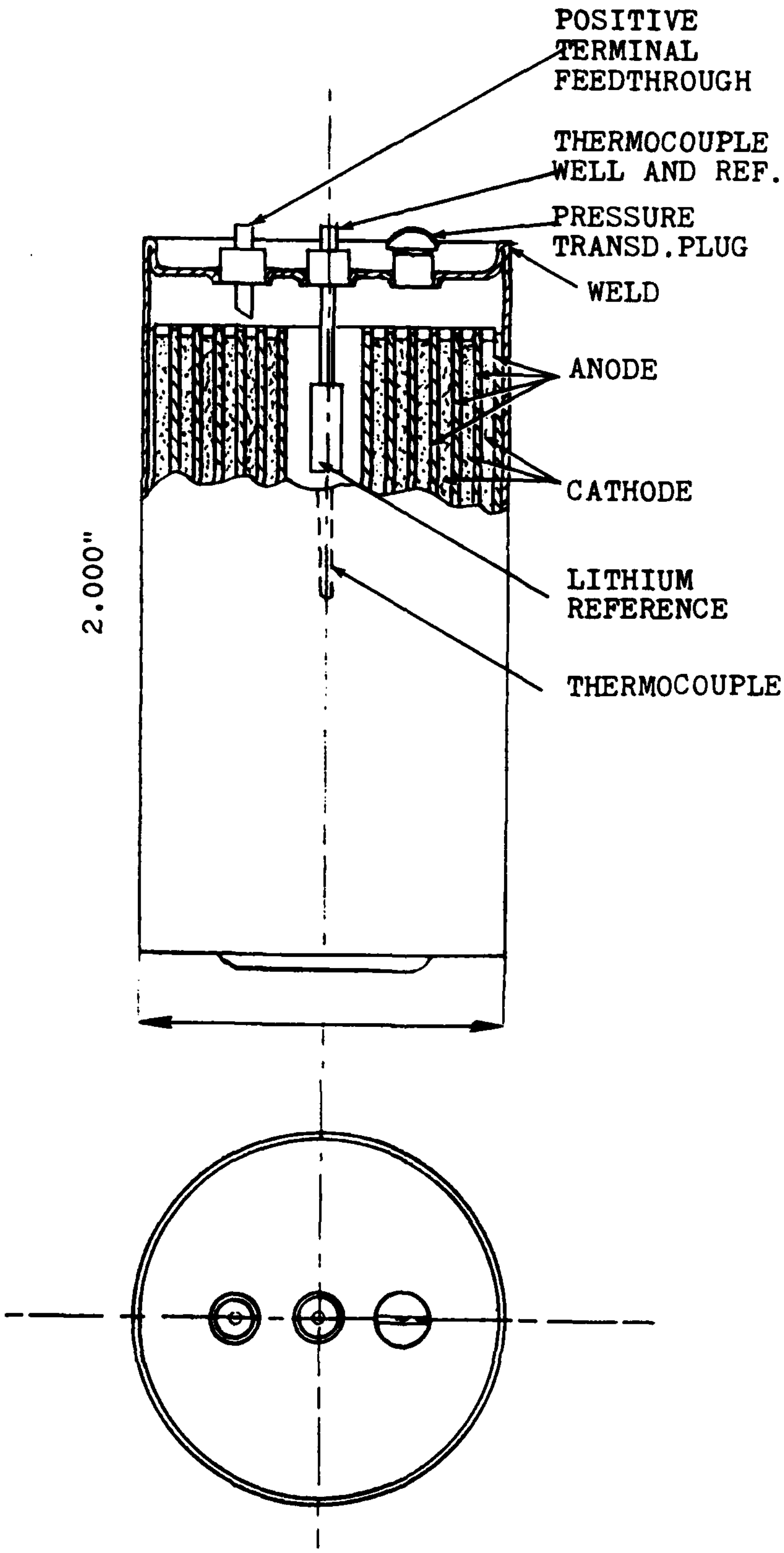

FIGURE 1. SCHEMATIC DIAGRAM OF THE INSTRUMENTED CELLS

TABLE 1. SPECIFICATIONS OF (DRY OR WET) Li/$SOCl_2$ CELLS

Spiral Design

	Lithium Limited		Cathode Limited	
	Li	C	Li	C
Length (in.)	14.75	13	11.5	10
Width (in.)	1.5	1.5	1.5	1.5
Thickness (in.)	.009 $\pm$ .001	.025 $\pm$ .003	.023 $\pm$ .001	.025 $\pm$ .003
Wt. (g)-Uncatalyzed	1.1 $\pm$ .15	4.3 $\pm$ .5	2.4 $\pm$ .2	3.3 $\pm$ .5
Wt. (g)-Catalyzed	1.1 $\pm$ .15	4.6 $\pm$ .5	2.4 $\pm$ .2	3.5 $\pm$ .5
Electrolyte Wt (g)	24.5 --	25	19.5 --	20
*Void Volume (ml.)	1.5 --	1.8	1.5 --	1.8

Disk Design

	Lithium Limited		Cathode Limited	
	Li	C	Li	C
Diameter (in.)	1.0	.875	1.0	.875
Width of Hole (in.)	.312	.250	.312	.250
Thickness (in.)	.006	.025	.020	.025
Wt. (g)-Uncatalyzed	1.1 $\pm$ .15	4.5 $\pm$ .25	2.7 $\pm$ .15	2.85 $\pm$ .25
Wt. (g)-Catalyzed	1.1 $\pm$ .15	4.5 $\pm$ .25	2.7 $\pm$ .15	2.85 $\pm$ .25
Electrolyte Wt (g)	22 --	23	18.5 --	19.5
*Void Volume (ml.)	1 --	1.5	1 --	1.5

40 ppm, expressed gravimetrically. The water content of the cells was measured by removing electrolyte samples from cells stored one week. The hydrolysis products present were analyzed using infrared spectroscopy and quantitatively measured against IR calibration curves prepared from samples of the dry electrolyte containing known amounts of water.

The cell tests were carried out in a hermetically sealed chamber as shown in Figure 2. The chamber, fitted with an O-ring seal cover and four compression springs, was designed to vent if the internal pressure of the chamber exceeded 50 psig. A stainless steel capillary tubing with ~0.5cc of void volume connects the pressure transducer located on the outside of the chamber to the cell inside.

Omega Engineering thermocouple connectors were used to connect the thermocouples from the cell to the stainless steel sheathed thermocouple wires. The sheathed wires were sealed with a compression fitting in the test chamber wall.

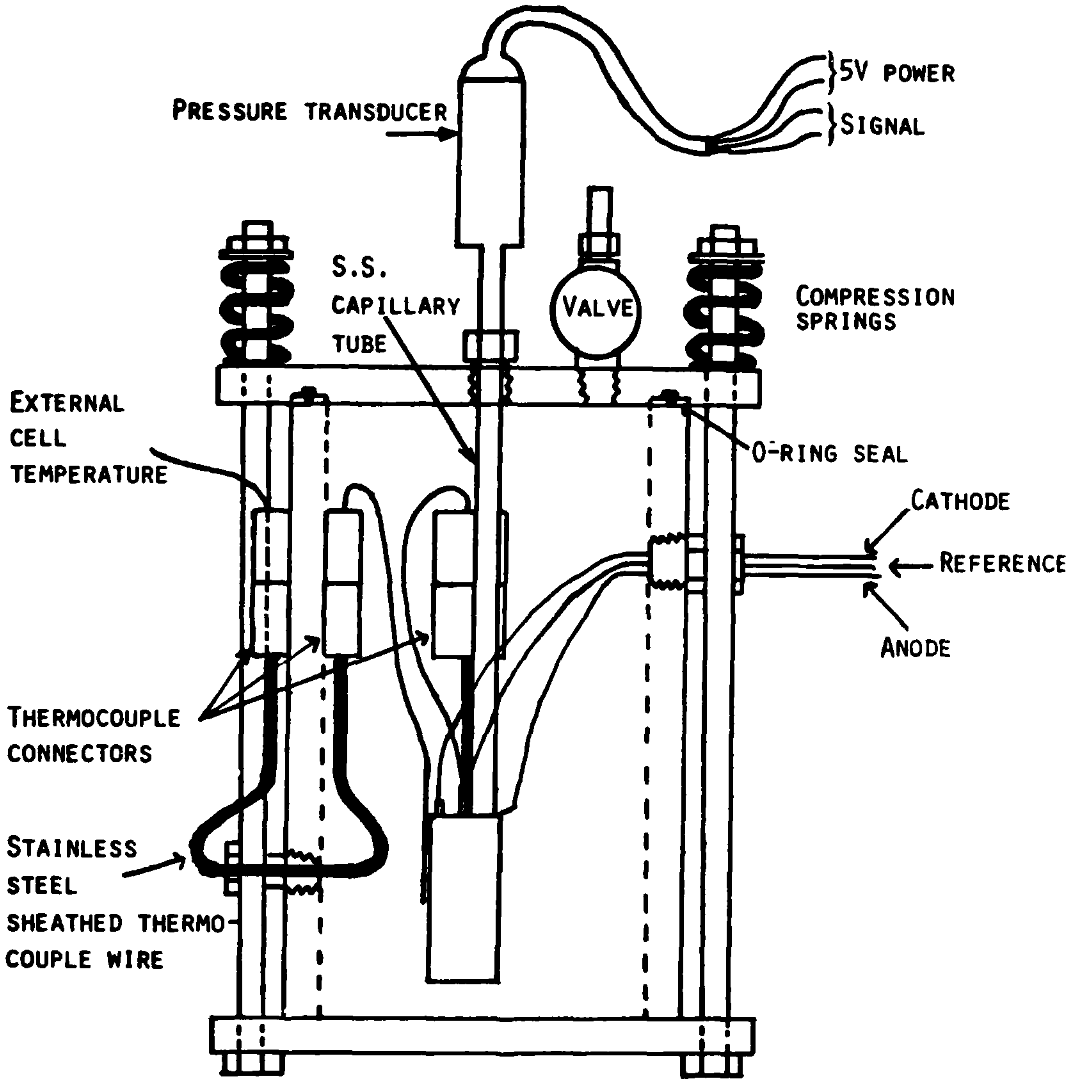

FIGURE 2. TEST CHAMBER FOR DISCHARGING $Li/SOCl_2$ CELLS

CHAPTER 3

RESULTS

BASELINE STUDY

A baseline study was carried out using two types of cathode limited cells to arrive at the optimum current density for testing. The cells were forced discharged and overdischarged to >150 percent of their capacities. The test data are shown in Table 2.

The type C_x cathode-limited cell contained a 26.7 cm x 3.8 cm x 0.08 cm lithium electrode weighing 3.90 g (15.1 Ah) with a total surface area of 203 cm^2. The carbon (2.90 g) cathode was 22.9 cm x 3.8 cm x 0.06 cm for a total surface area of 174 cm^2. The cells contained 18 g of 1.8 M $LiAlCl_4$ in $SOCl_2$ (6.5 Ah).

The type C_y cathode limited cell specifications are listed in Table 1. The lithium electrode had 9.3 Ah capacity and a total surface area of 222 cm^2. The carbon cathode had 193 cm^2 total surface area. These cells contained 19.5 g of 1.8M $LiAlCl_4$ in $SOCl_2$ (7.04 Ah).

Low Rate Discharge (25°C)

Both C_x and C_y type cells were discharged and overdischarged at 200 mA current (1mA/cm^2). They exhibited similar discharge behavior as illustrated in Figure 3. After overdischarge each cell was analyzed. Gas phase IR analysis revealed only $SOCl_2$ and SO_2 present. There was no visual evidence of Li being plated onto the carbon.

Moderate Rate Discharge (25°C)

Cell C_y-2, discharged at 5.2 mA/cm^2, experiences an increased anode polarization at the higher current as shown in Figure 4.

High Rate Discharge (25°C)

The discharge curve for cell C_x-2 discharged at 11.5 mA/cm^2 is shown in Figure 5. The internal cell temperature reached 45°C during discharge and increased to 110°C after 30 minutes into overdischarge.

TABLE 2. PERFORMANCE IN BASELINE STUDY OF CATHODE-LIMITED CELLS

Cell Number	Discharge Current, A	Current Density (mA/cm^2)	Capacity to 0.0V, A-hr	Results
C_y-1	0.2	1.0	4.3	Cathode-limited, no hazards.
C_x-1	0.2	1.1	4.3	Cathode-limited, no hazards.
C_y-2	1.0	5.2	3.0	Cathode-limited, no hazards.
C_x-2	2.0	11.5	2.0	Cathode-limited, no hazards.
C_y-3	2.0[1]	10.4	3.8	Cathode-limited. Cell vented during overdischarge.
C_y-4[2]	2.0	10.4	3.1	Cathode-limited. Cell vented during overdischarge.
C_x-3	2.0	11.5	1.7[3]	Anode-limited, no hazards.

[1]Due to power supply malfunction, current was not constant throughout discharge. Actual capacity was more like Cell Cy-4.

[2]Cell contained 500 ppm of water, i.e., wet cell.

[3]Cell discharged at -13°C.

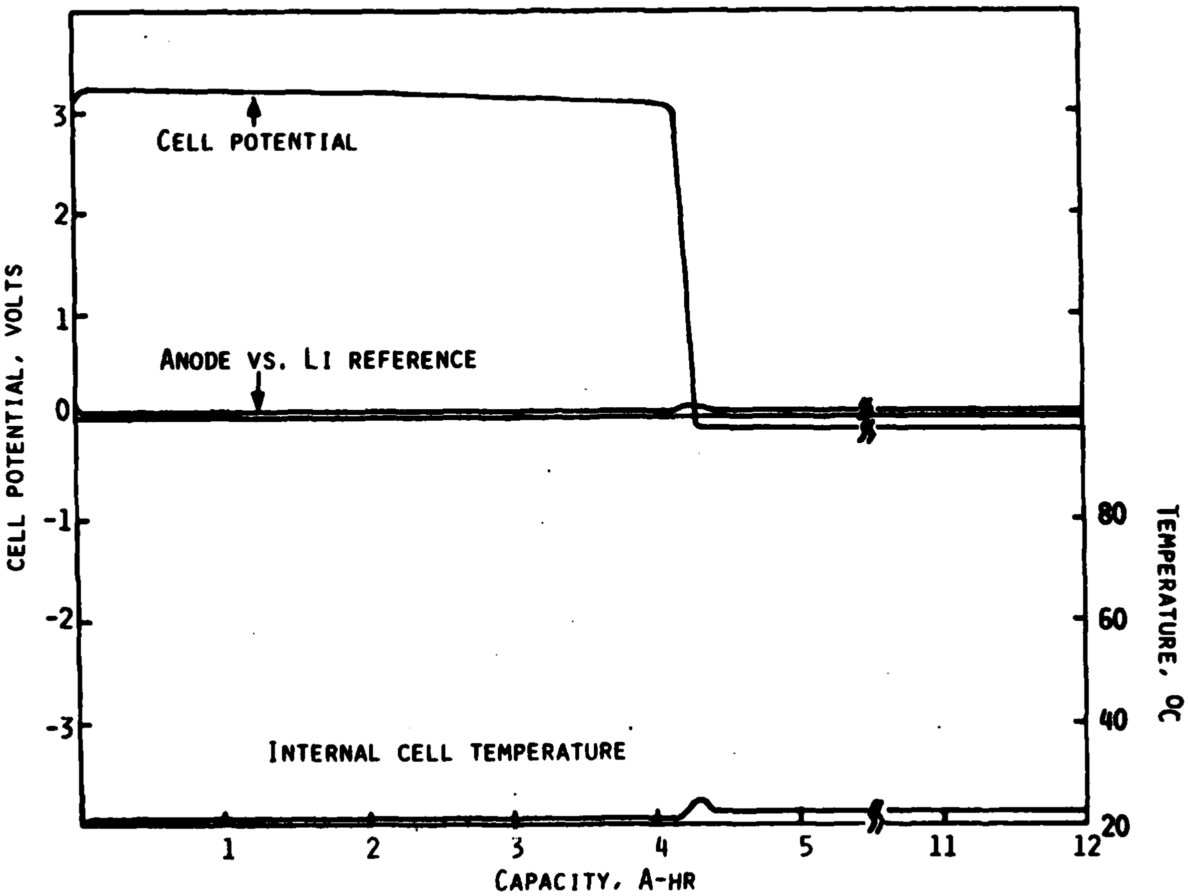

FIGURE 3. DISCHARGE DATA FOR CELL C_y-1 AT 0.2A

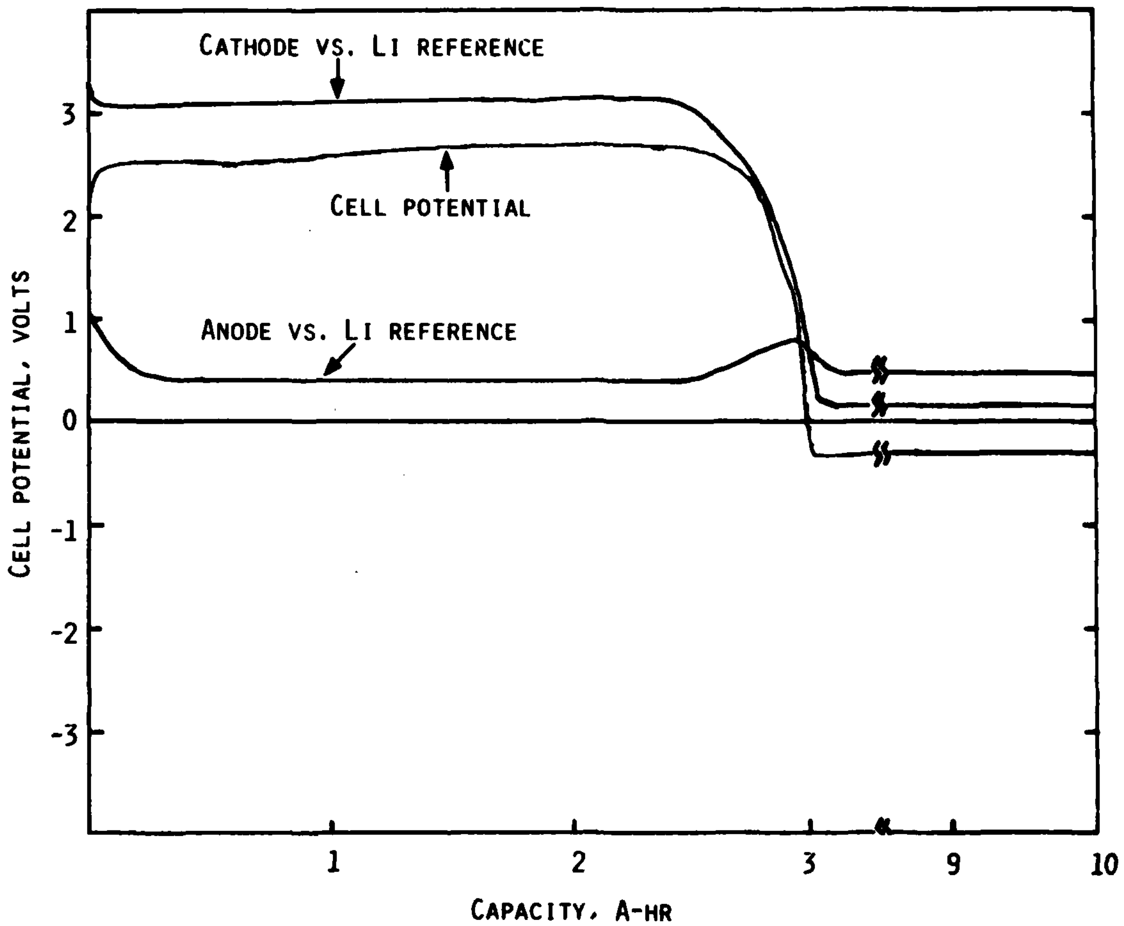

FIGURE 4. DISCHARGE DATA FOR CELL C_y-2 AT 1.0A

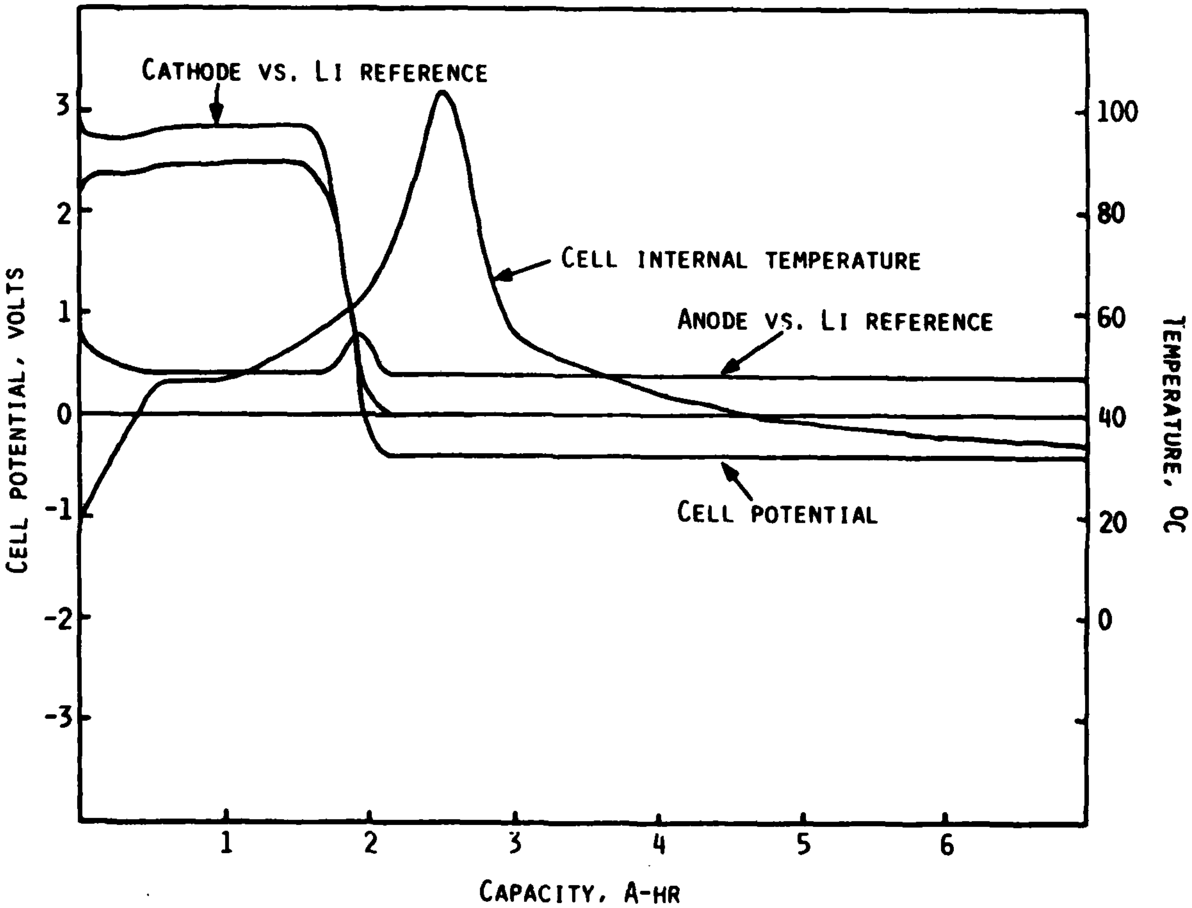

FIGURE 5. DISCHARGE DATA FOR CELL C_x-2 AT 2.0A

Cell C_y-3, discharged at 10.4 mA/cm^2 reached 60°C during discharge and rose to 145°C during the first 30 minutes of reversal. Although the cell vented, it was not clear when the venting occurred. The data are shown in Figure 6.

Cell C_y-4, containing 500 ppm water, was also discharged at the 2.0 amp rate (10.4 mA/cm^2) into reversal.

Gas samples from both C_y-3 ("dry") and C_y-4 ("wet") were compared using IR analysis and are shown in Figures 7 and 8, respectively. In addition to $SOCl_2$ and SO_2 both show HCl present as evidenced by the multiple peaks from 2700 to 3000 cm^{-1}. The peaks at 2040 and 2060 cm^{-1} resemble COS. The "wet" cell showed enhancement of the band at 550 cm^{-1} and a band now appears at 670 cm^{-1} with a shoulder near 685 cm^{-1}. Recent IR studies on the solution of flooded $SOCl_2$ cells showed somewhat similar bands appearing at 694 cm^{-1} during discharge and 665 cm^{-1} during reversal. The 694 cm^{-1} band was attributed to the presence of water and was observed to decrease after discharge while the 665 cm^{-1} band increased upon reversal.[2]

High Rate Discharge (-15°C)

Figure 9 shows the voltage-temperature data for cell C_x-3 discharged into reversal at a 2.0 amp rate at -15°C. No venting occurred.

DISCUSSION

Type C_y cathode-limited cells were selected for further testing.

Analysis of the discharge data for type C_x cathode-limited cells occasionally showed considerable anode polarizations as illustrated in Figure 9. In fact, the end of cell life of cell C_x-3 can be considered anode-limited at 2.0 A (11.5 ma/cm^2) at -15°C. The decrease in cell potentials towards end of life of this cell is associated with a positive polarization of the Li electrode. The cathode shows negative polarizations soon after the polarization in the anode potentials. The lithium electrode was anodically polarized, presumably initiated by a nonhomogeneous current distribution towards the end of discharge and/or a concentration polarization due to crystals forming on the surface of the lithium. This phenomenon is somewhat similar to the "voltage delay" due to film passivation but in this case it appears to be triggered anodically due to high current densities and/or low temperature environments.

This phenomenon may have an important bearing on the safety hazards of $Li/SOCl_2$ cells. It has been shown that cathode or Li-limited cells that have lithium remaining (anode limited) at the end of cell life may be hazardous during forced overdischarge.[3] In actuality, we now know that at high current densities, especially at low temperatures, both cathode and Li-limited cells can undergo anodic passivation and become anode limited, i.e., a $Li/SOCl_2$ cell may become anode limited despite the presence of significant amounts of Li on the anode.

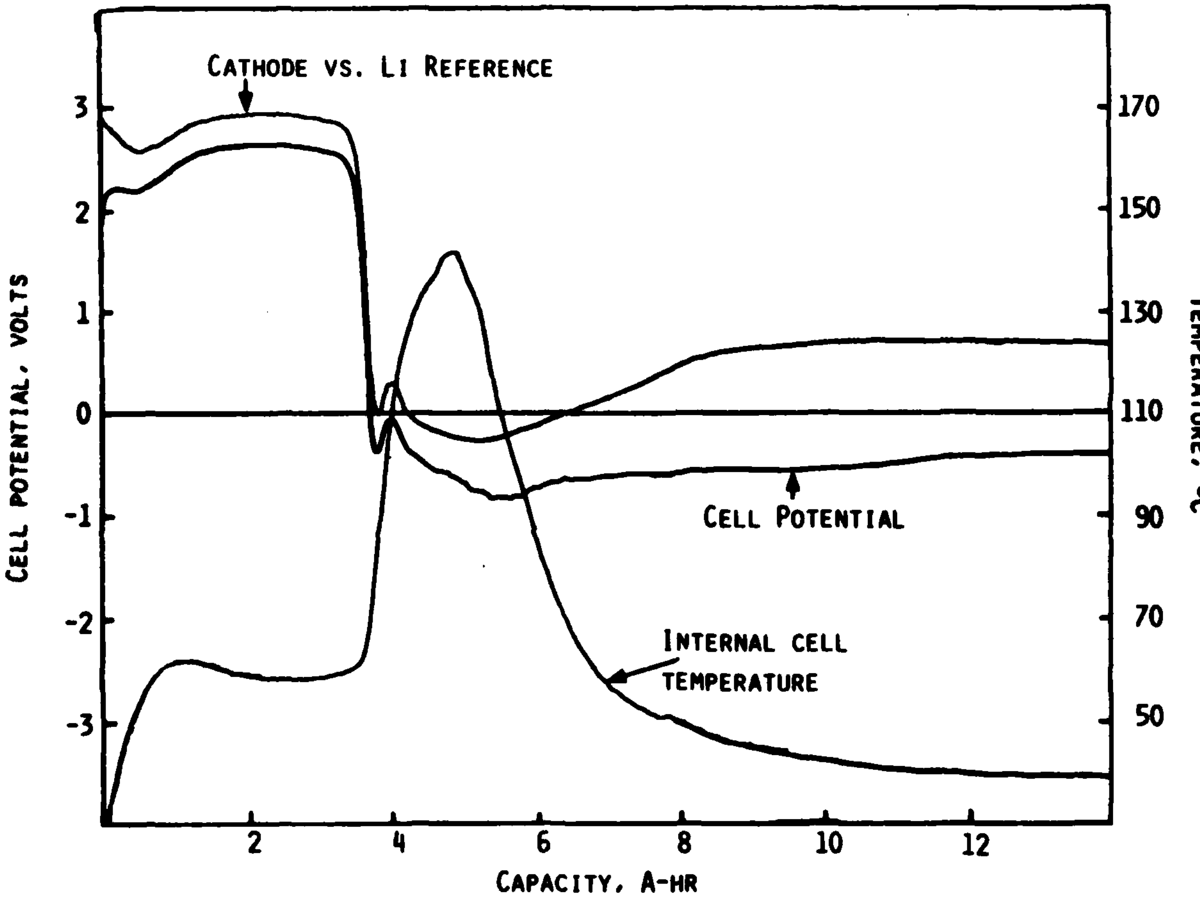

FIGURE 6. DISCHARGE DATA FOR CELL C_y-3 AT 2.0A

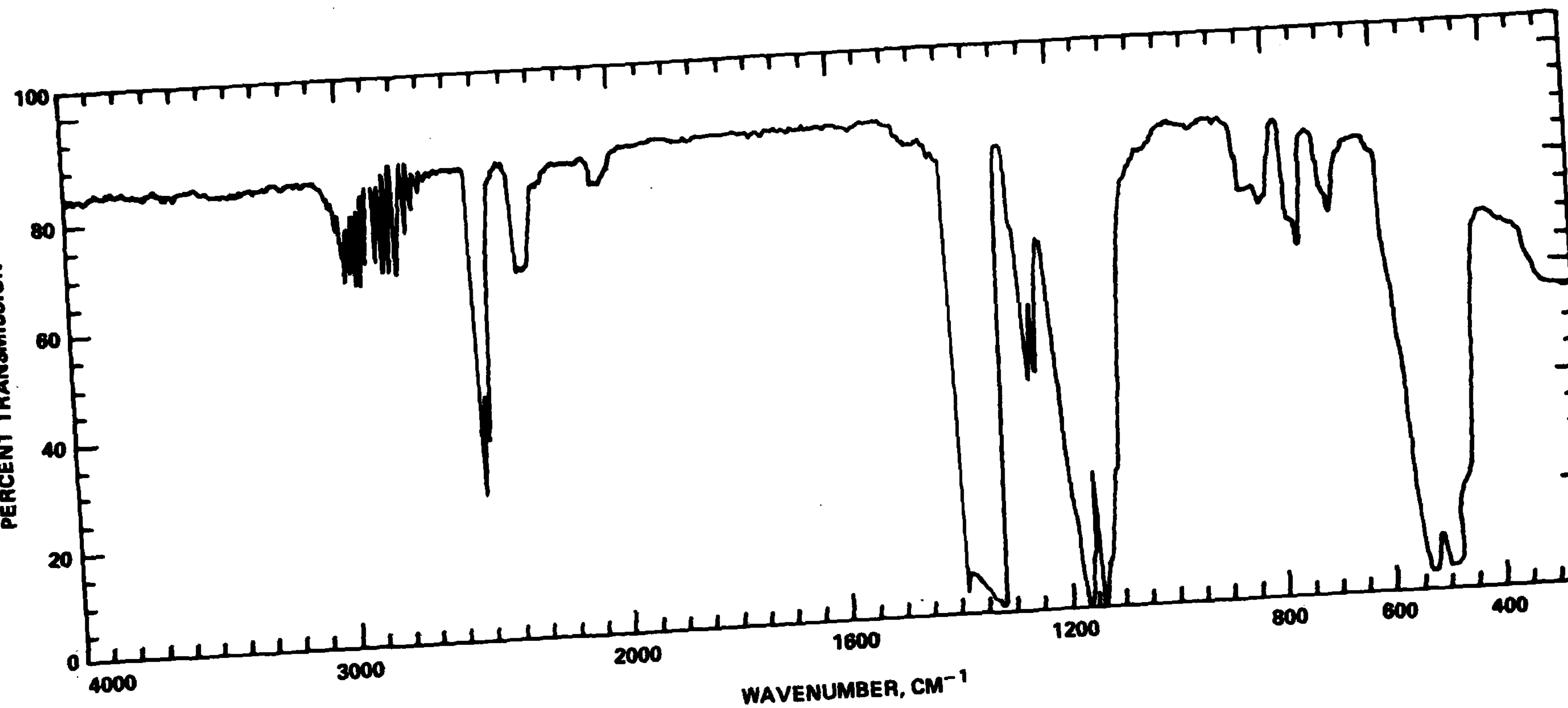

FIGURE 7. IR SPECTRUM OF GAS FROM "DRY" VENTED CELL C_y-3

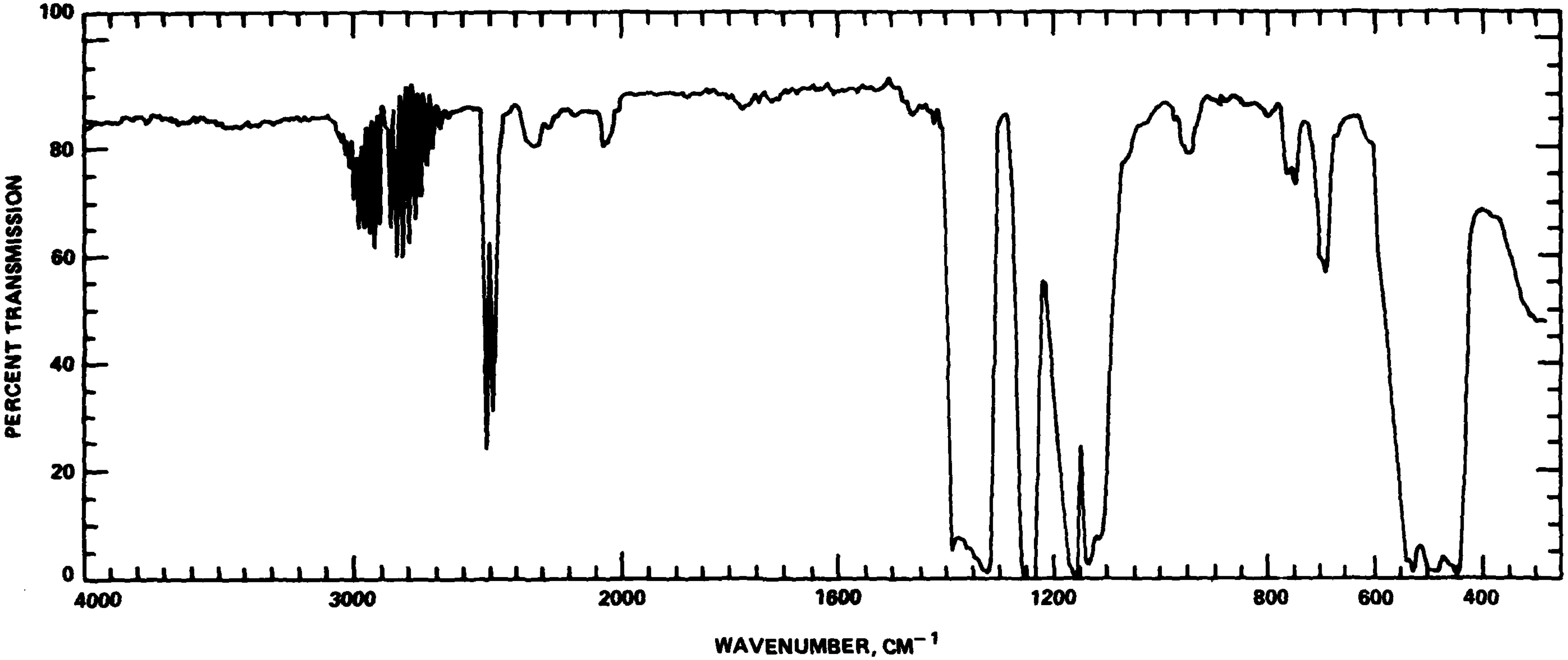

FIGURE 8. GAS IR OF PRODUCTS FROM "WET" CELL C_y-4

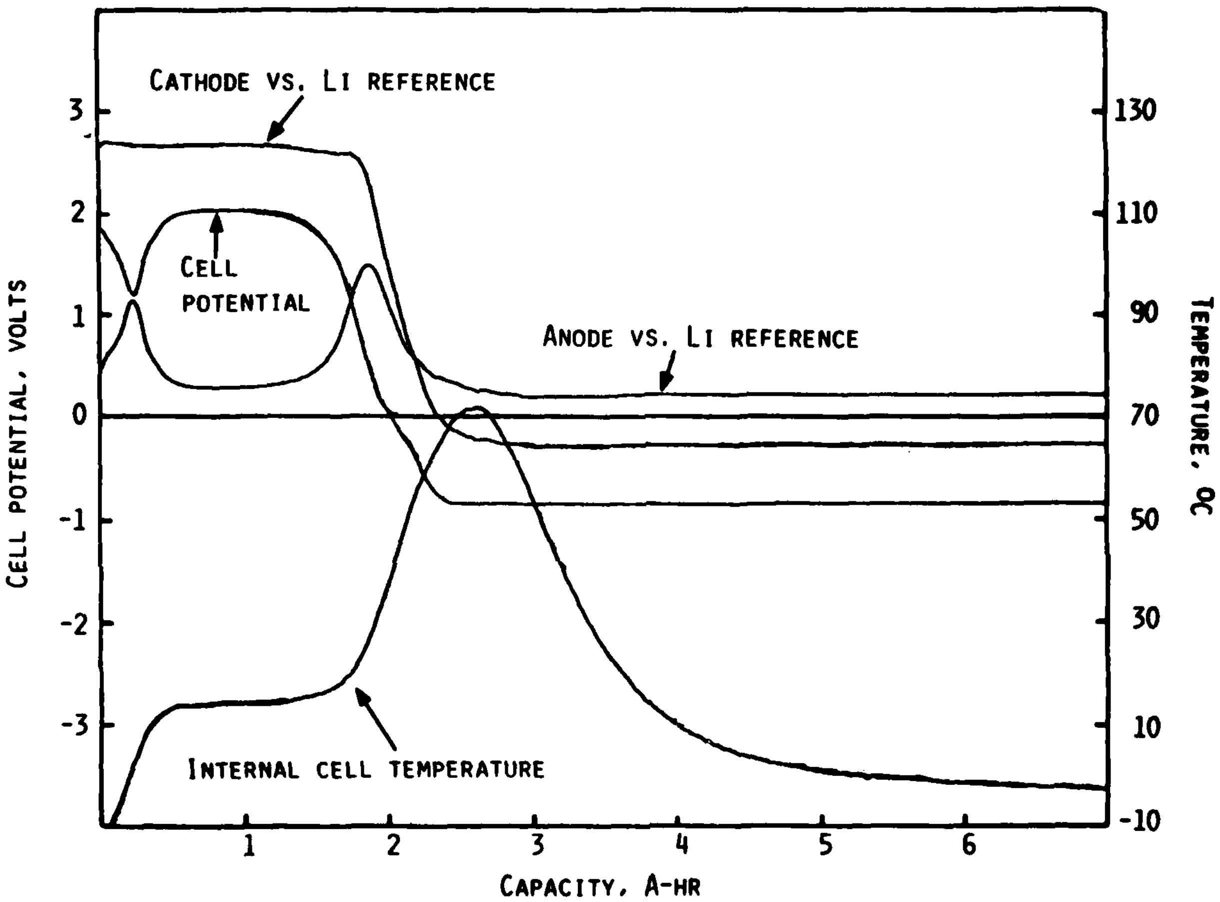

FIGURE 9. DISCHARGE DATA FOR CELL C_x-3 AT 2.0A, AND -15°C

DISCHARGE AND OVERDISCHARGE OF LITHIUM-LIMITED CELLS

The Li-limited cells were discharged at 1.0, 2.0 and 3.0 amp rates, corresponding to 4, 8 and 12 mA/cm^2 in order to determine the optimal high current density for use in this study. The cell performance is shown in Table 3.

The cell discharged at 1.0 amp (Figure 10) showed no significant change in temperature (maximum 44°C) or pressure during overdischarge. The performance of the cell discharged at 2.0 amp is shown in Figure 11. The end of discharge was caused by anode limitation. After the test, the cell was disassembled. There was no lithium remaining on the anode grid. Cell A-3, discharged at the 3.0 amp rate apparently became cathode limited at this higher current. The discharge curve is shown in Figure 12. After three hours in overdischarge, the temperature showed a sharp increase. The cell vented. The pressure plot showed only a modest increase before dropping off. The reaction apparently happened too fast to record.

The IR spectrum of the gases vented from cell A-3 is shown in Figure 13. The spectrum shows HCl, SO_2 and $SOCl_2$. There are also strong absorptions due to other materials. The doublet at 2060 and 2080 cm^{-1} is COS. The doublet at 1530 and 1545 cm^{-1} is that of CS_2.

Dry Cells

Dry cells, containing 50 ppm water labeled A-D, were discharged and over-discharged at currents of 0.2A and 2.0A at 20°C and -12°C. The performance data are tabulated in Table 4. The cells discharged at 20°C showed only modest increases in temperature and pressure. Typical data at 2.0A is illustrated for cell A-D-8 in Figure 14. The cells discharged at -12°C were still Li-limited and yielded the same capacity at the 2.0A rate (Figure 15) as that obtained at room temperature. The mid-discharge voltage was slightly lower and typically showed a substantial temperature increase at the end of discharge, accompanied by a slight increase in pressure. It should be noted that the thermocouple response at -12°C was poor resulting in an inaccurate pressure reading at this temperature.

Chemical analysis of the cells tested at 20°C and -12°C at 2.0A gave similar results. IR analysis of the gases showed only $SOCl_2$ and SO_2. IR analysis of the cathode showed no absorptions. Spot tests on the cathode for sulfur-oxy compounds were negative.

Since anode polarizations to positive potentials may lead to oxidation processes producing chlorine, gases from cells A-D-11 and A-D-12 were tested for chlorine by passing them through a dry ice-acetone trap to first remove $SOCl_2$ and then collected in a liquid nitrogen trap. The gases were then bubbled through a starch and KI solution. Both tests were negative.

A small amount of salt was extracted from the electrolyte of cell A-D-11. It was recrystallized and a KBr pellet was made for IR analysis. The spectrum is shown in Figure 16. Water bands appear at 3400 and 1650 cm^{-1} due to absorption by the $LiAlCl_4$. The broad bands at 3050, 2400, 830 and 495 cm^{-1} can be assigned to $LiAlCl_4$. Four unidentified peaks at 970, 1070, 1200 and 1330 cm^{-1} are present in the spectrum where many sulfur-oxy compounds absorb.

TABLE 3. PERFORMANCE IN BASELINE STUDY OF LITHIUM-LIMITED CELLS

Cell Number	Discharge Current, A	Current Density* (mA/cm^2)	Capacity to 0.0V, A-hr	Results
A-1	1.0	5.2	3.3	Cell Li-limited, showed no hazard on forced overdischarge.
A-2	2.0	10.4	3.0	Cell Li-limited, showed no hazard on forced overdischarge.
A-3	3.0	15.6	3.2	Cathode-limited, cell vented on forced overdischarge.

*Current density based on cathodes from previous lithium-limited cells.

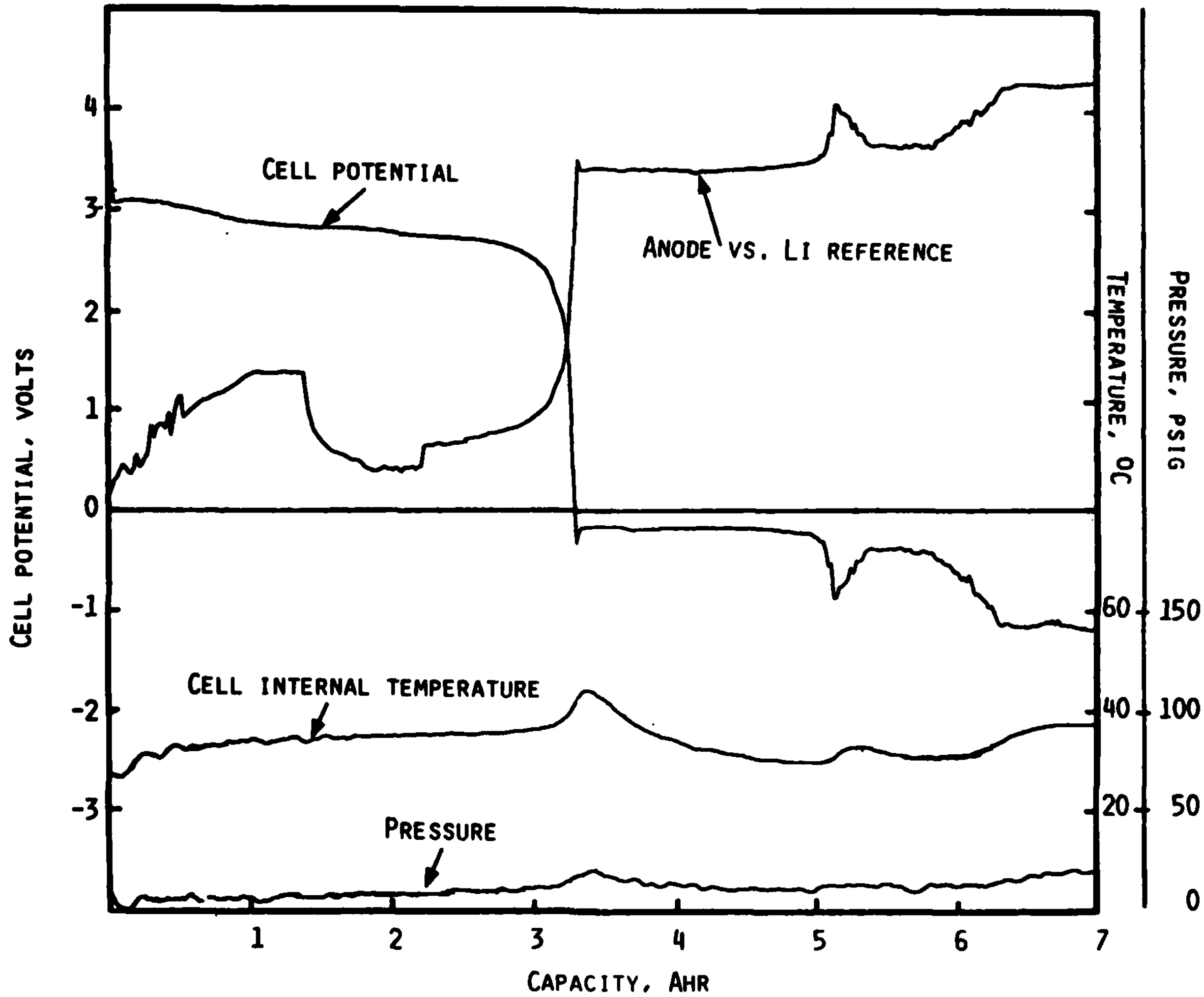

FIGURE 10. DISCHARGE DATA FOR CELL A-1 AT 1.0A

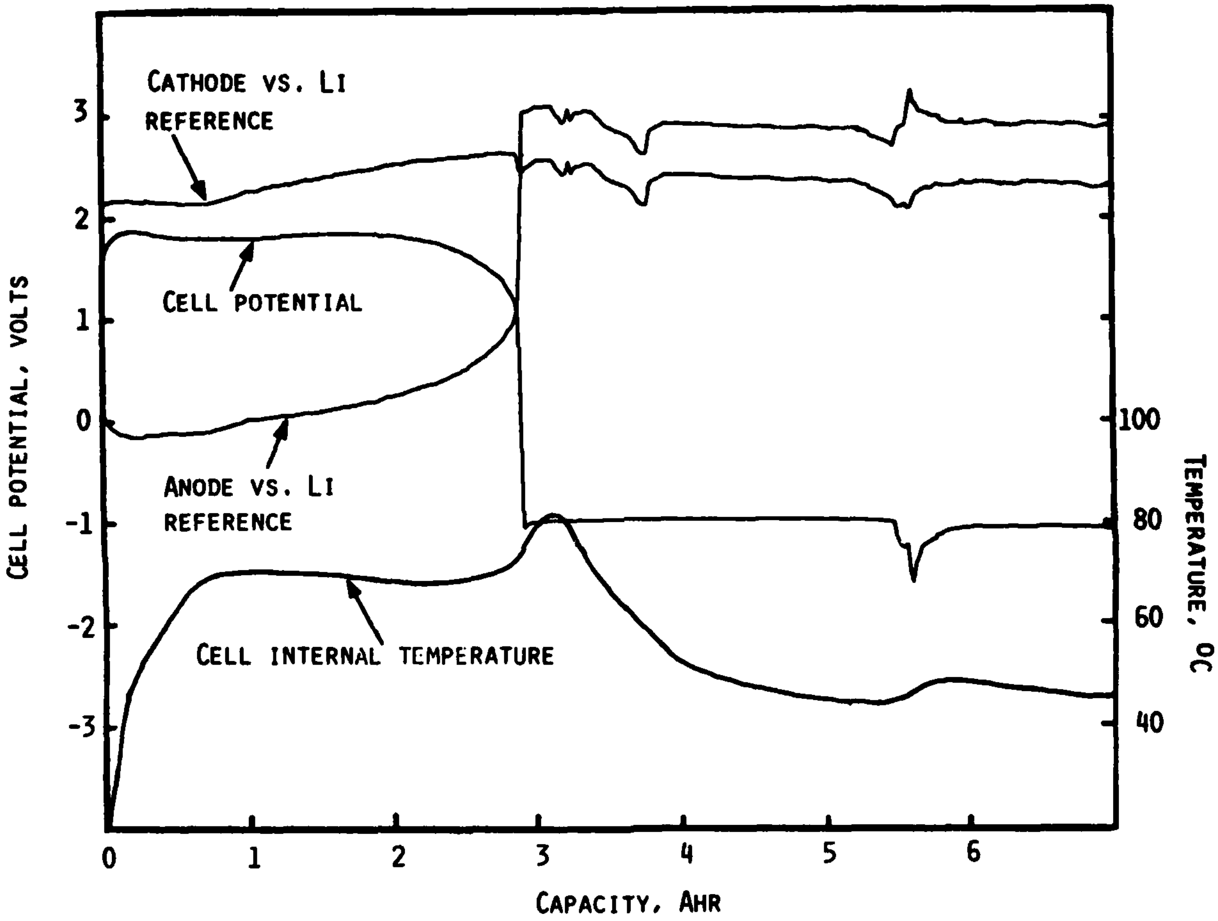

FIGURE 11. DISCHARGE DATA FOR CELL A-2 AT 2.0A

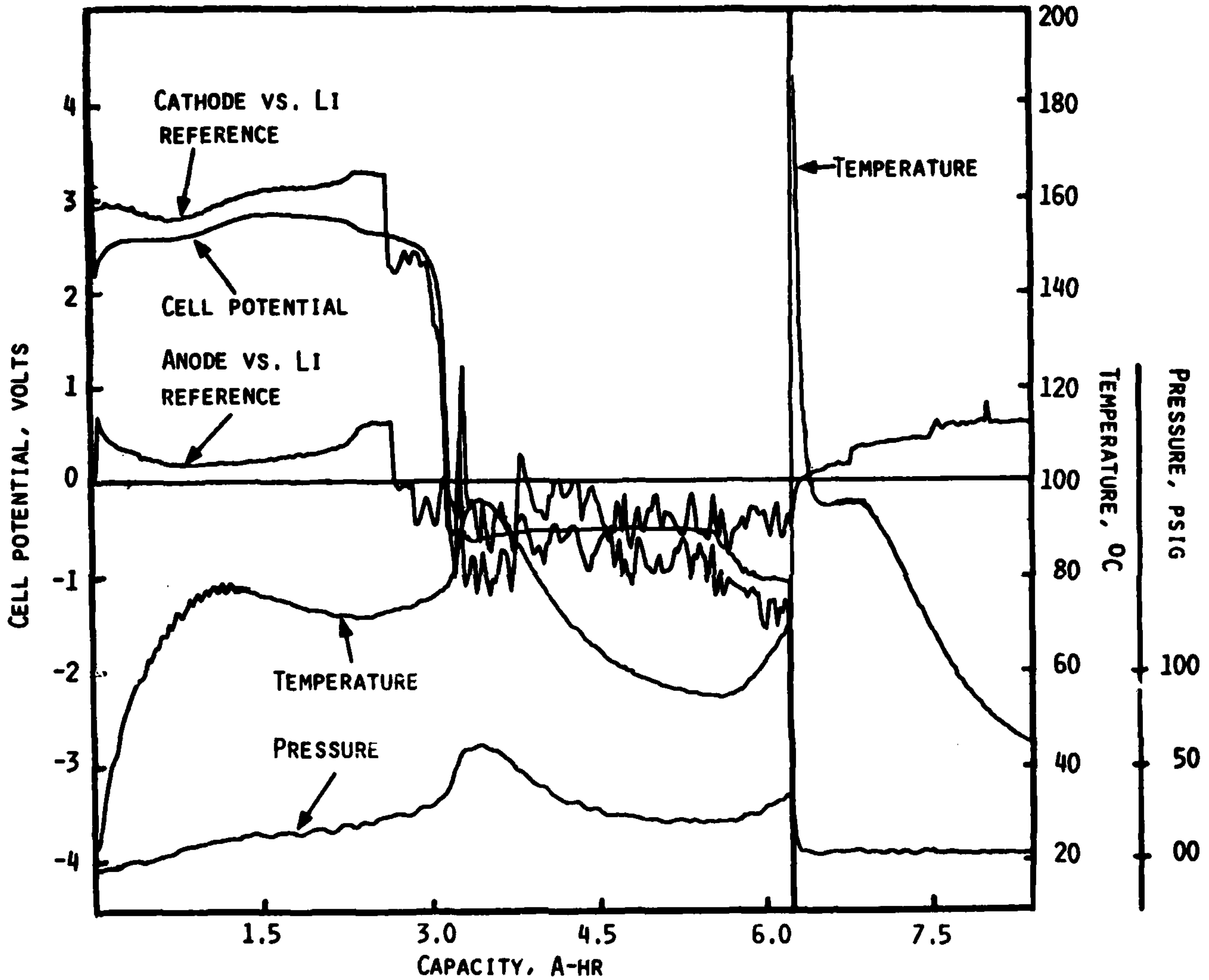

FIGURE 12. DISCHARGE DATA FOR CELL A-3 AT 3.0A

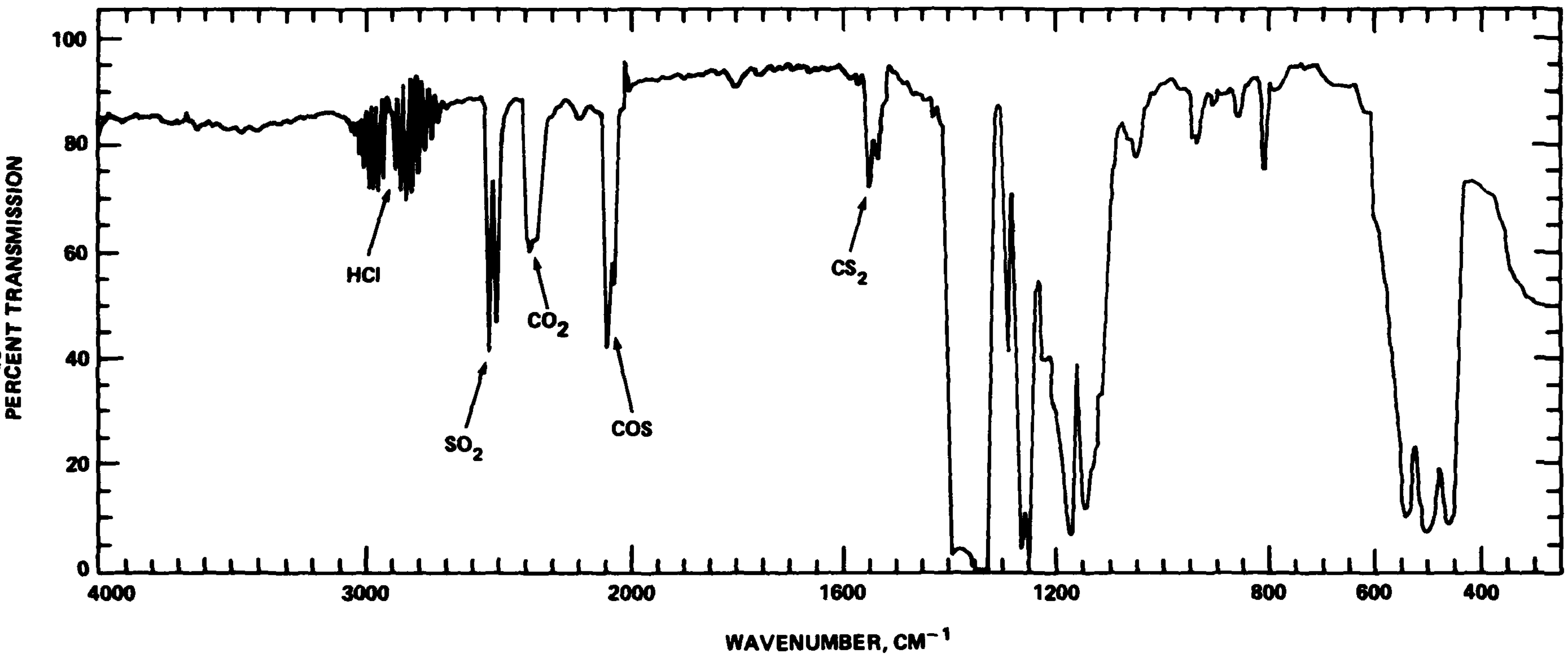

FIGURE 13. GAS IR SPECTRUM FROM CELL A-3

TABLE 4. PERFORMANCE OF LITHIUM-LIMITED DRY CELLS

Cell* Number	Discharge Current (A)	Temperature (°C)	Capacity to 0.0V (Ah)
A-D-4	2.0	20	3.00
A-D-5	2.0	20	2.85
A-D-6	2.0	20	1.29
A-D-7	2.0	20	3.05
A-D-8	2.0	20	2.90
A-D-9	0.2	20	3.00
A-D-10	2.0	-12	2.81
A-D-11	2.0	-12	3.00
A-D-12	2.0	-12	2.90
A-D-13	0.2	-12	2.70

*Cells showed no hazardous behavior. Cell A-D-6 showed very low capacity. Cell A-D-11 exhibited no polarization of the anode.

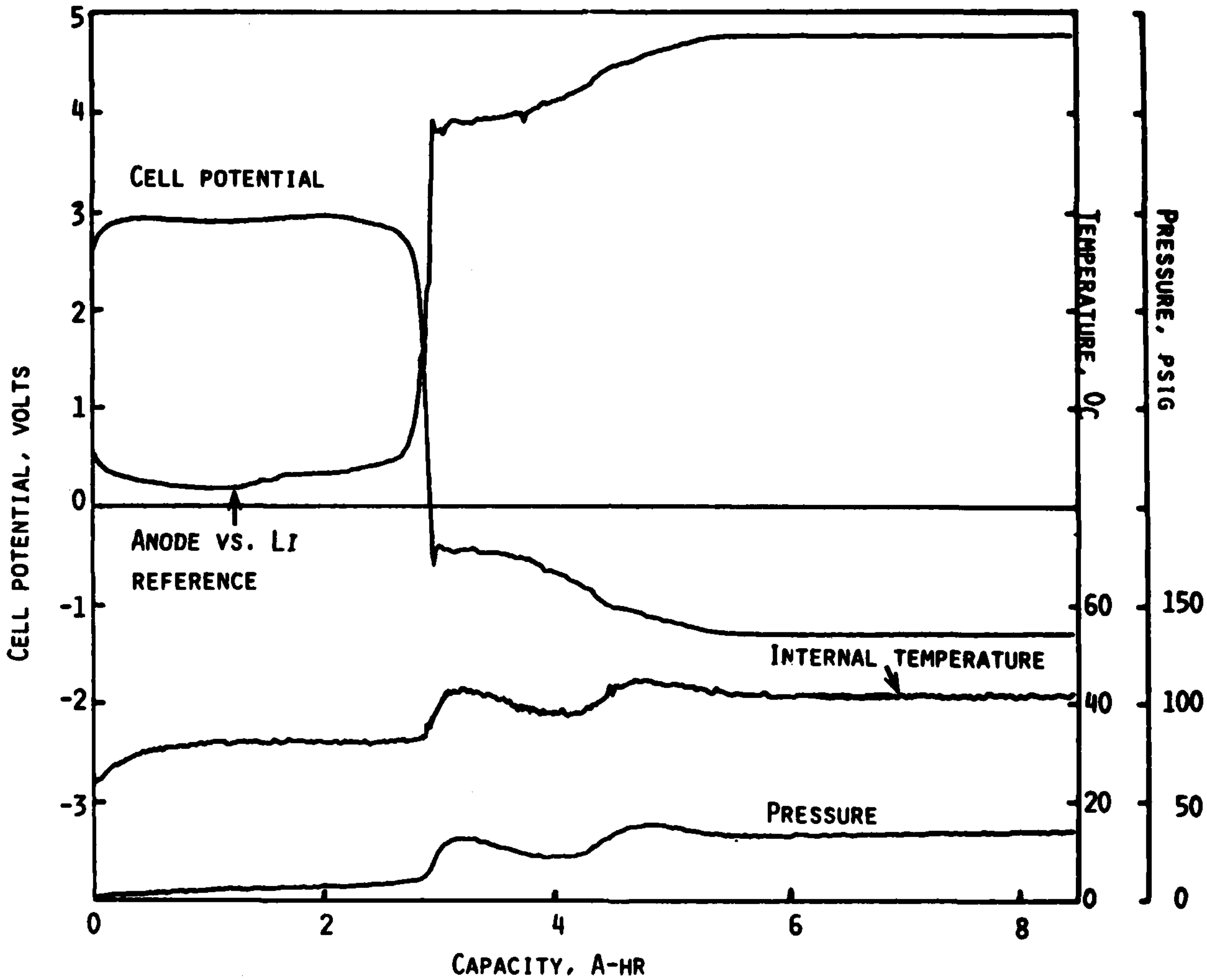

FIGURE 14. DISCHARGE DATA FOR CELL A-D-8 AT 2.0A

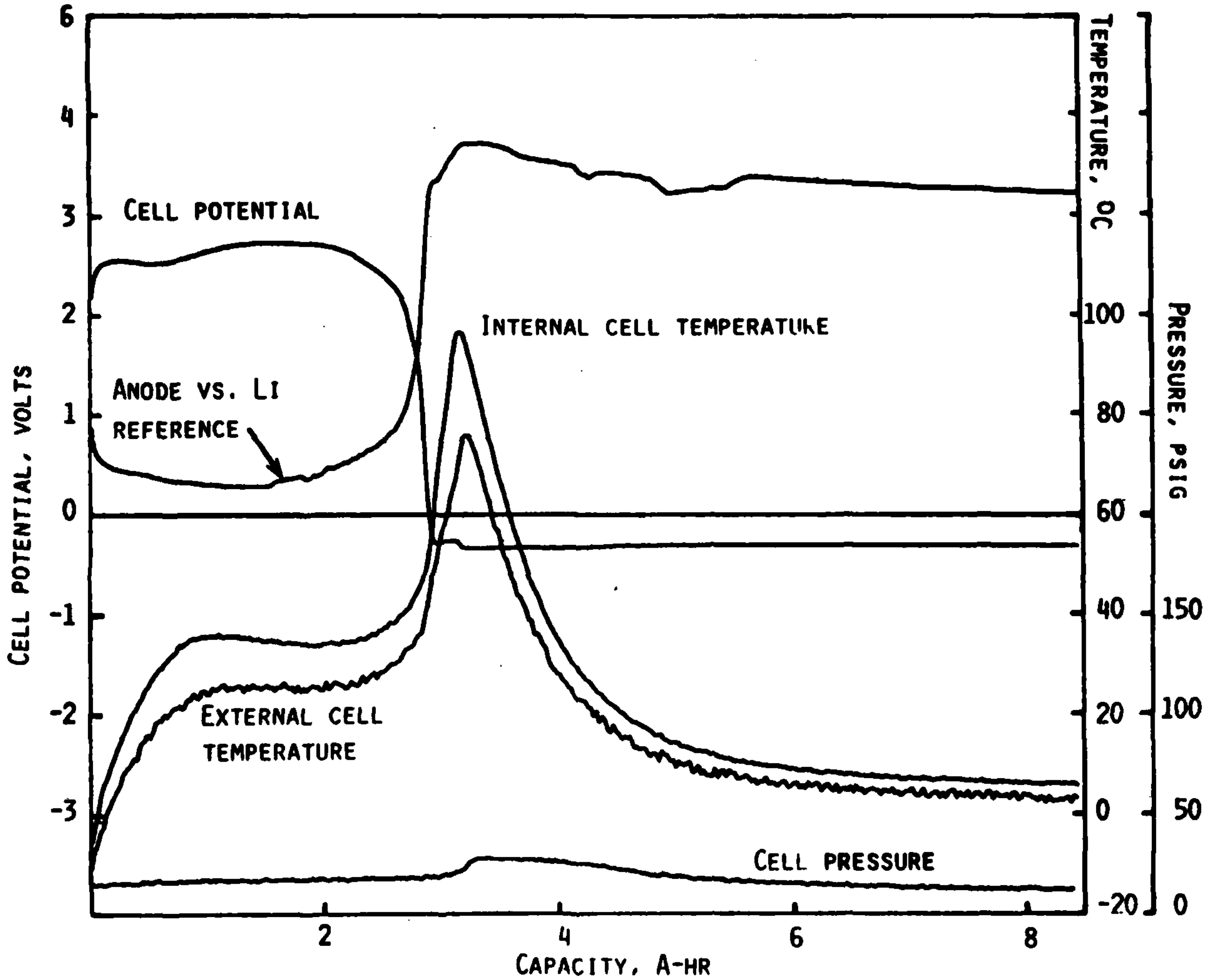

FIGURE 15. DISCHARGE DATA FOR CELL A-D-12 AT 2.0A AND –12°C

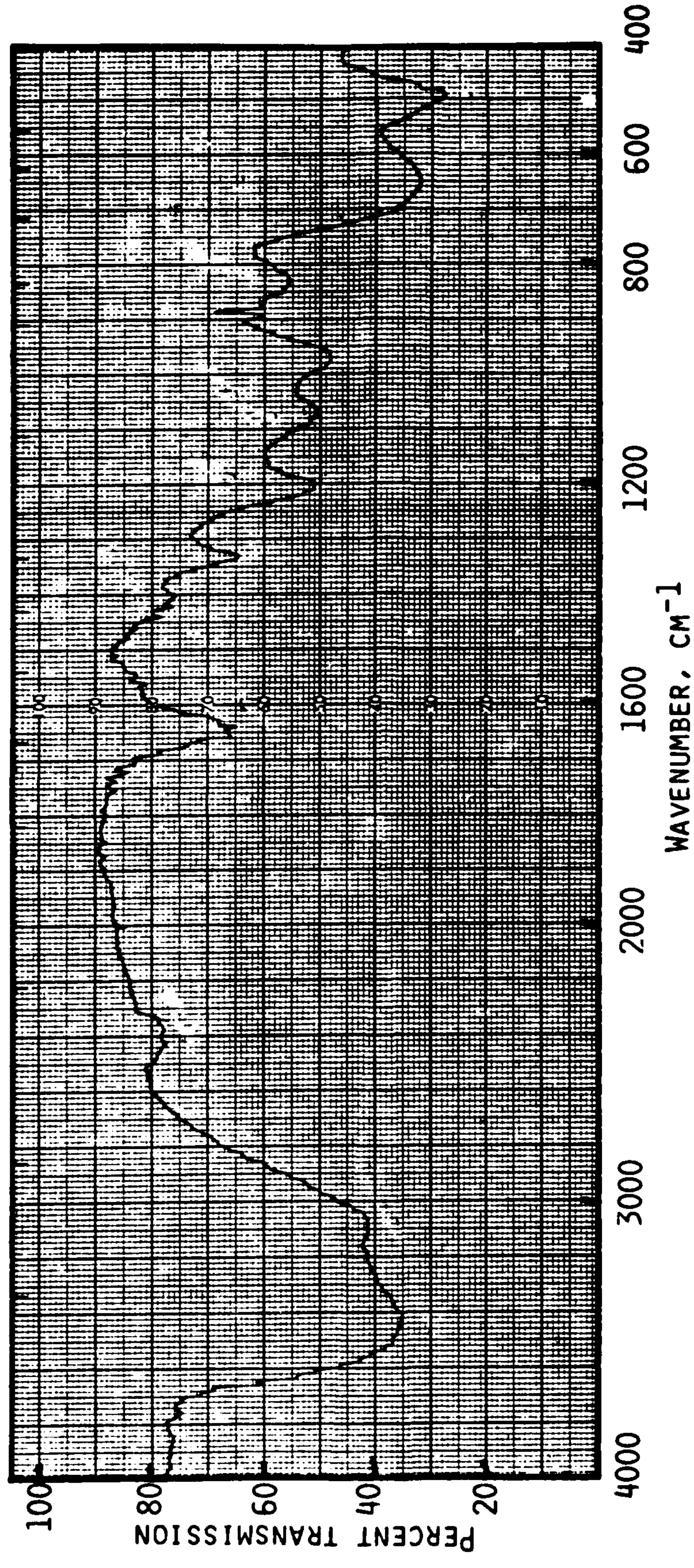

FIGURE 16. IR SPECTRUM OF SALT EXTRACTED FROM CELL A-D-11

Wet Cells

Li-limited wet cells, labelled A-W, were discharged at 0.2 and 2.0A rates at 20°C and -12°C. The performance data are summarized in Table 5. The discharge curve for cell A-W-18 is shown in Figure 17. The IR spectrum of the gases produced from cell A-W-18 is shown in Figure 18. The multiple peaks at 2900 cm^{-1} are due to HCl. An IR spectrum of solids extracted from cell A-W-15 revealed the same peaks present in Figure 16.

Typical performance data of cells discharged at 2.0A at -12°C are illustrated by cell A-W-21 in Figure 19. The internal temperature reached 56°C while the external temperature reached 35°C. Figure 20 shows the IR spectrum of the gases from cell A-W-21. Only $SOCl_2$ and SO_2 are present with no HCl.

DISCUSSION

The Li-limited wet cells generally showed lower capacity than the corresponding dry cells. Low temperature discharging appeared to have a greater effect on the performance of the wet cells. A greater percent temperature rise occurs at the end of discharge in the low temperature cells. Though anodic potentials reached voltages capable of forming chlorine, no chlorine gas could be isolated. It is possible that the Cl_2 generated may be reduced at the carbon cathode, or it may combine with SO_2 to form SO_2Cl_2 with little of it accumulating in the cell. Chlorine exhibits substantial solubility in $SOCl_2/LiAlCl_4$ and this solubility may be another reason for no Cl_2 in the gas phase. Soluble solids extracted from the electrolyte for IR analysis indicate S-O bonded reaction products. Presumably, as the $SOCl_2$ is reduced solvates of $LiAlCl_4.xSOCl_2$ and $LiAlCl_4.ySO_2$ form. The broad bands observed near 2400, 1330, 1200, 970, 830 and 495 cm^{-1} agree well with the 2394, 1330, 1162-1223, 966-974, 825 and 498 cm^{-1} bands found in the IR spectra of $LiAlCl_4.2SOCl_2$ and $LiAlCl_4.3SO_2$ previously reported.[4] The absorptions observed at 1330 and 1070 cm^{-1} may be due to $Li^+[SO_2, SO_2Cl_2]$ $AlCl_4^-$.[1,2] The formation of this latter compound could account for the lack of free Cl_2 in the gas phase of the cells.

UNCATALYZED CATHODE-LIMITED CELLS

Dry Cells at 20°C

Table 6 summarizes the performance data for cathode limited dry $Li/SOCl_2$ cells, labeled C-D, discharged at 0.2A and 2.0A at 20 C. Although substantially high temperatures and pressures were reached during the tests, none of the cells vented. Cell C-D-9 was discharged without a pressure transducer to determine if it changed the cell behavior. This cell also did not vent. A few cells which had the internal thermocouple placed in the cell so that it was not residing in the middle of the cell but closer to the top of the can recorded a higher temperature maximum from the external thermocouple. Typical performance data for cells discharged at 0.2A and 2.0A are shown in Figures 21 and 22, respectively.

TABLE 5. PERFORMANCE OF LITHIUM-LIMITED WET CELLS

Cell* Number	Discharge Current (A)	Temperature (°C)	Capacity to 0.0V (Ah)
A-W-14	2.0	20	2.51
A-W-15	2.0	20	2.80
A-W-16	2.0	20	2.63
A-W-17	2.0	20	2.59
A-W-18	2.0	20	2.60
A-W-19	0.2	20	2.71
A-W-20	2.0	-12	2.40
A-W-21	2.0	-12	2.43
A-W-22	2.0	-12	2.29
A-W-23	0.2	-12	2.74

*Cells showed no hazardous behavior. Cell A-W-15 exhibited no polarization of the anode.

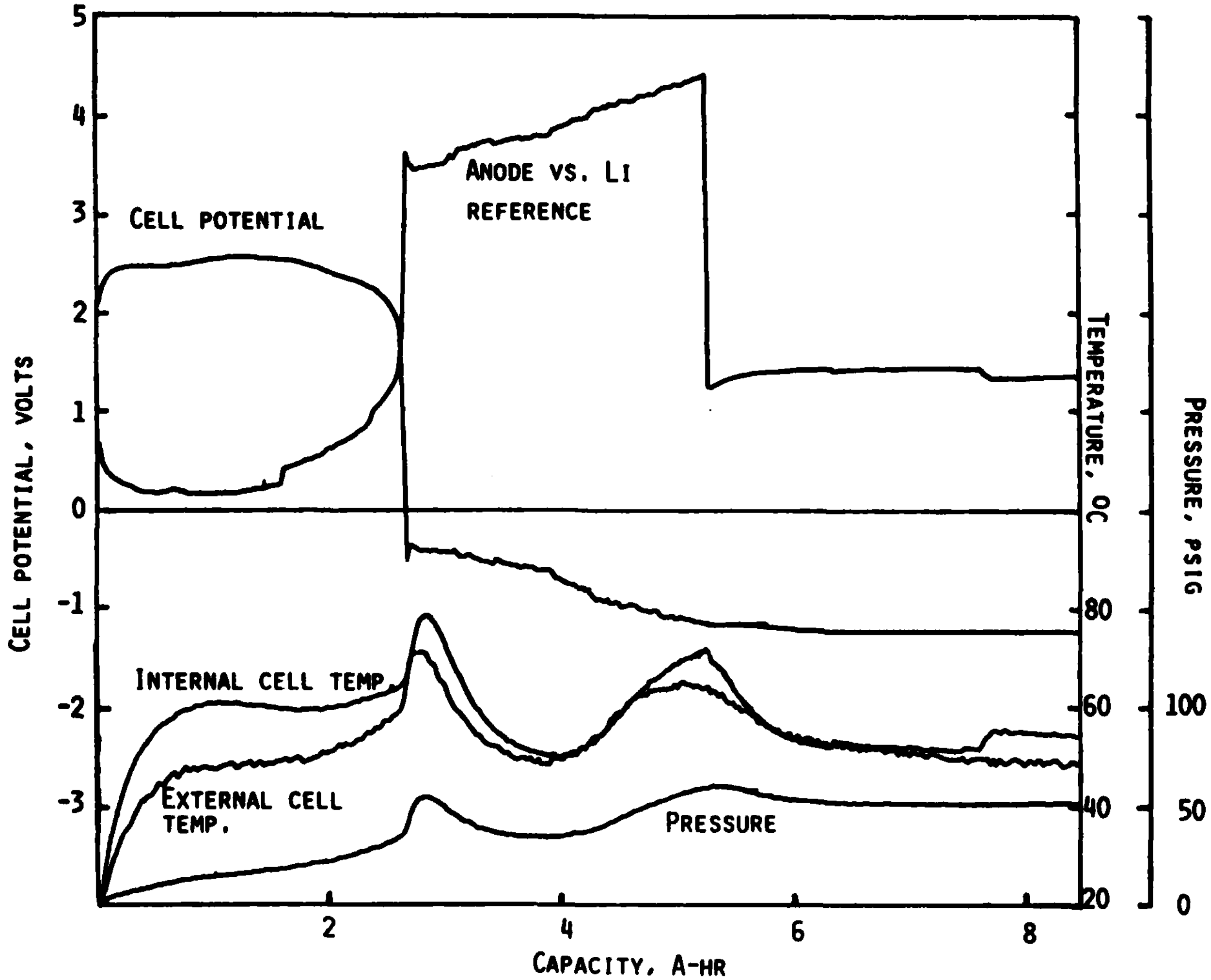

FIGURE 17. DISCHARGE DATA FOR CELL A-W-18 AT 2.0A

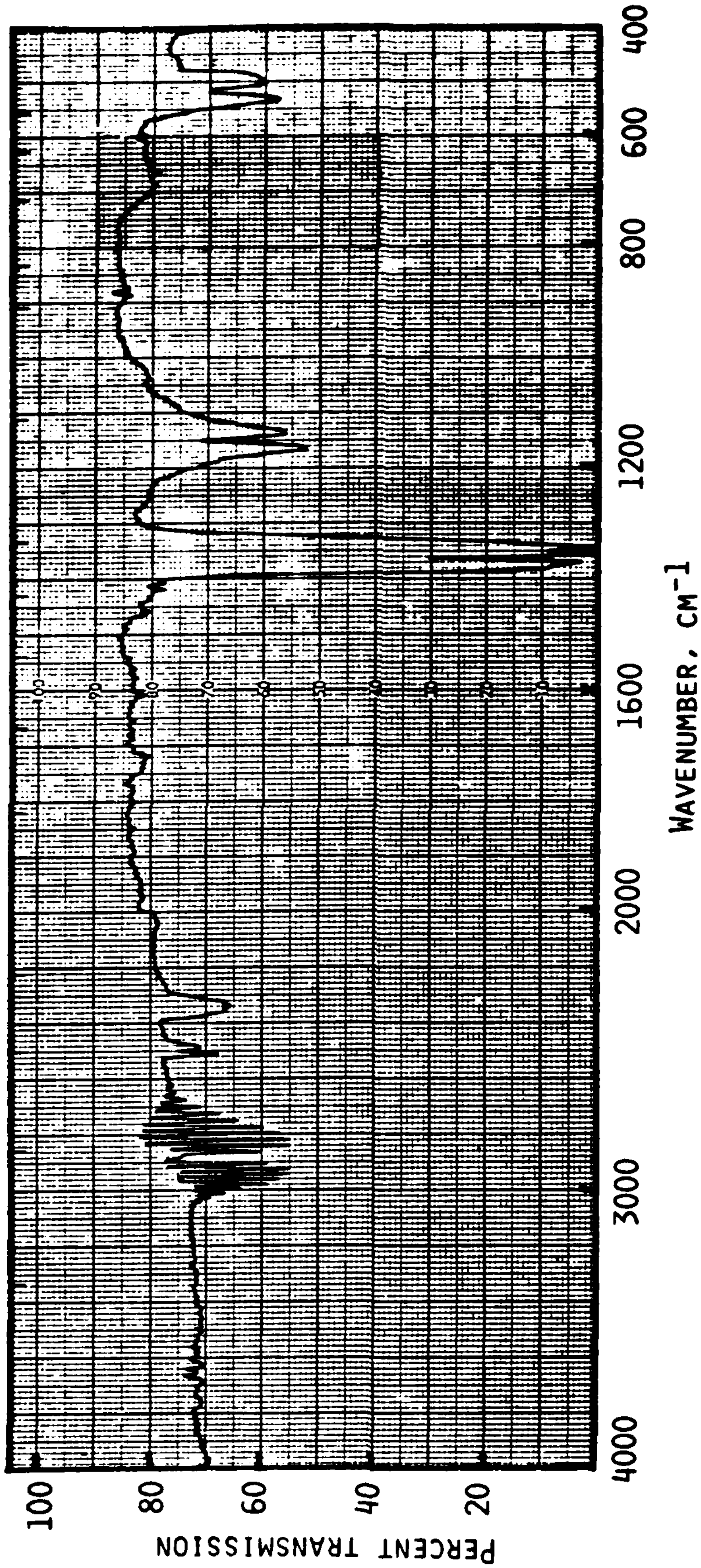

FIGURE 18. GAS IR SPECTRUM FROM CELL A-W-18

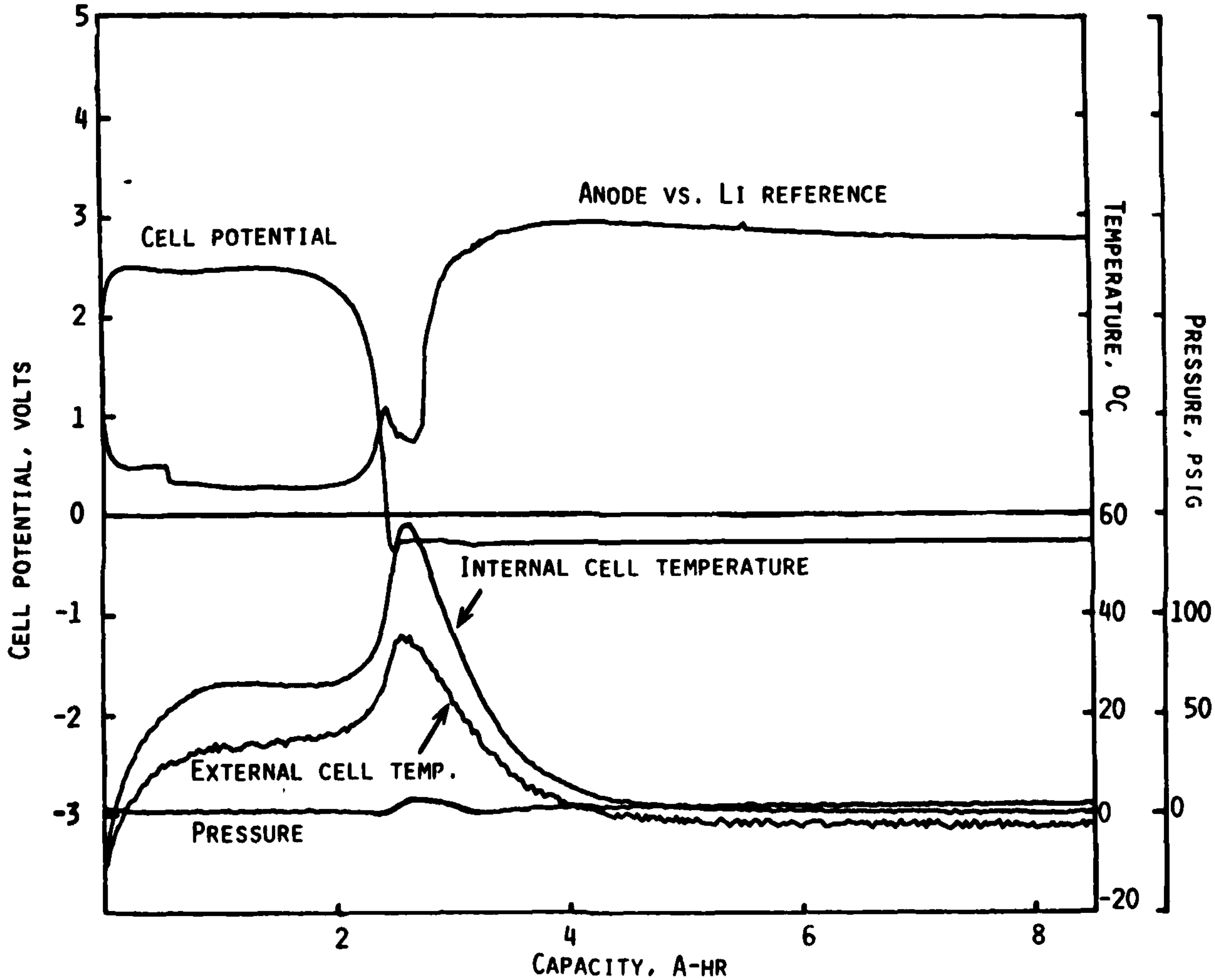

FIGURE 19. DISCHARGE DATA FOR CELL A-W-21 AT 2.0A AND −12°C

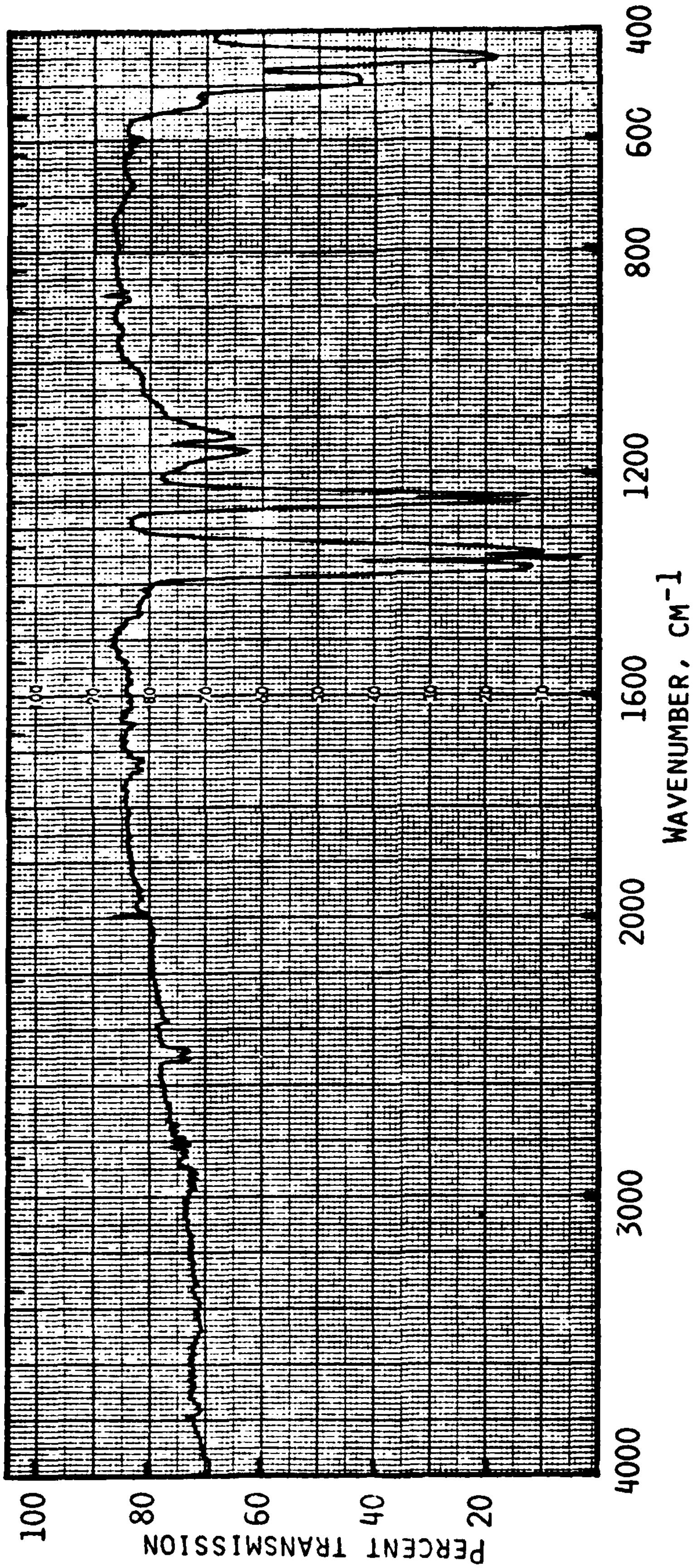

FIGURE 20. IR SPECTRUM OF GASES FROM CELL A-W-21

TABLE 6. PERFORMANCE OF CATHODE-LIMITED DRY CELLS AT 20°C

Cell* Number	Discharge Current (A)	Current Density (mA/cm^2)	Capacity to 0.0V (A-hr)	Maximum Temp. (°C)	Maximum Pressure (psig)
C-D-1	0.2	1.0	4.42	31	7
C-D-2	0.2	1.0	5.02	-	7
C-D-3	0.2	1.0	5.15	-	23
C-D-4	2.0	10.4	3.66	97	142
C-D-5	2.0	10.4	3.90	99	145
C-D-6	2.0	10.4	3.32	93	125
C-D-7	2.0	10.4	3.72	121	146
C-D-8	2.0	10.4	3.36	116	131
C-D-9	2.0	10.4	3.24	89	-

*Cells showed no hazardous behavior. The external temperature was higher in cells 4, 5 and 9. Cell 9 had no pressure transducer.

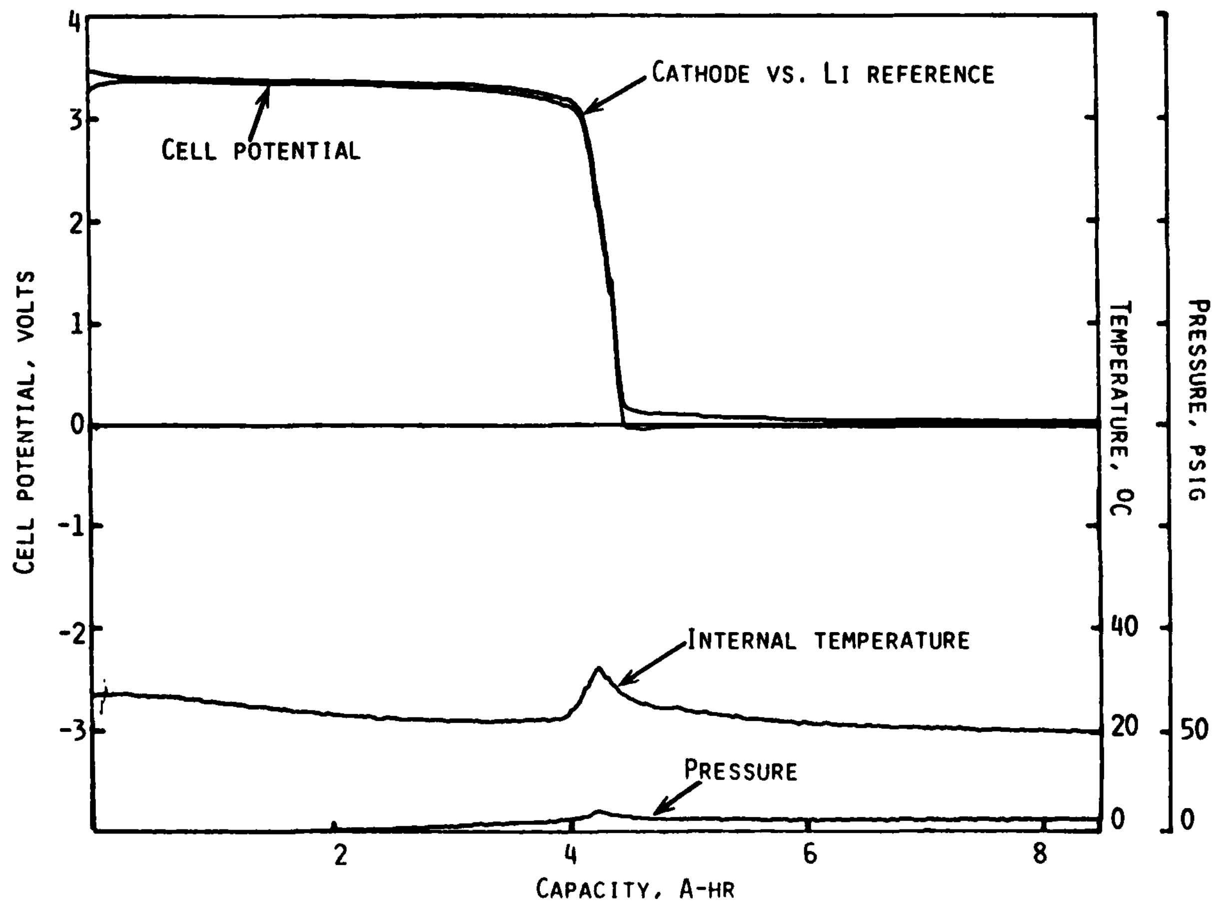

FIGURE 21. DISCHARGE DATA FOR CELL C-D-1 AT 0.2A

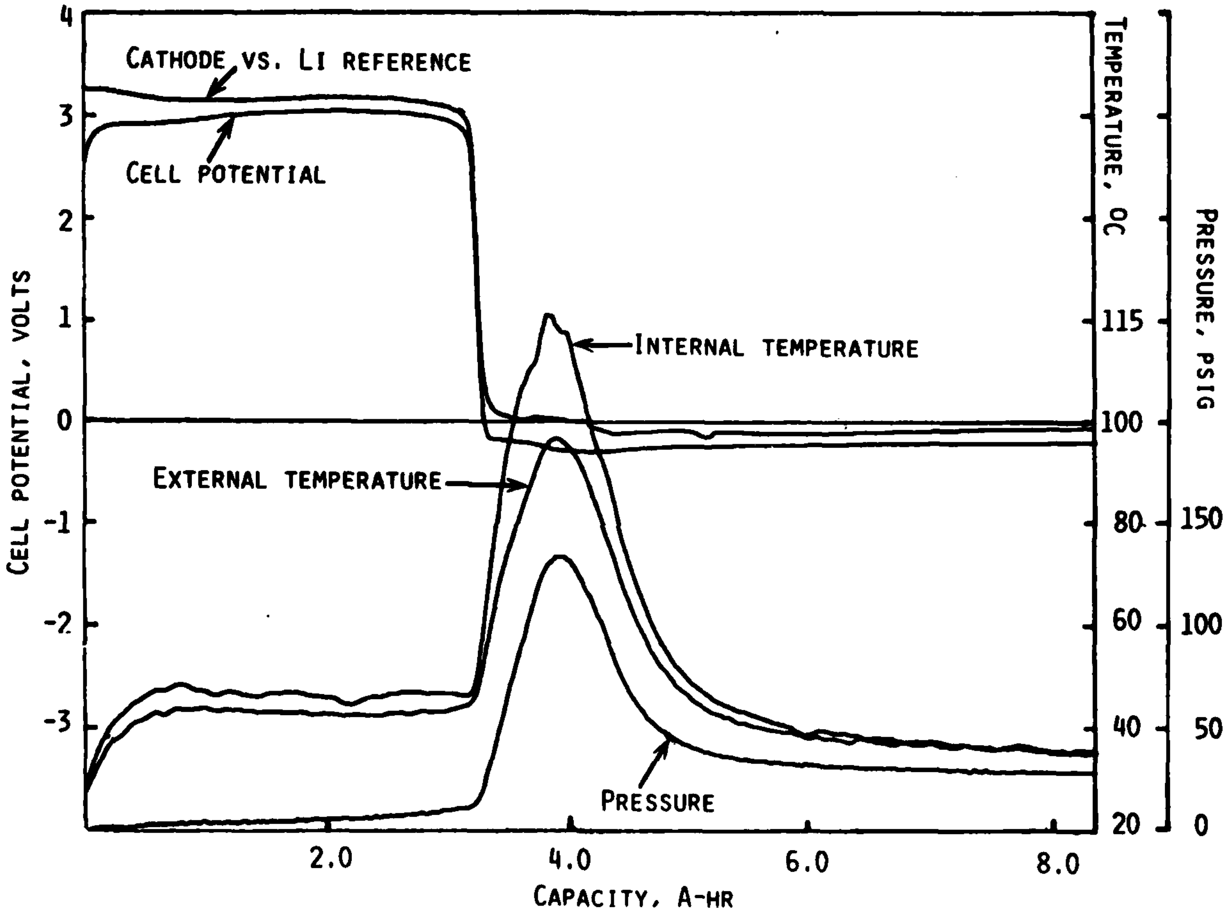

FIGURE 22. DISCHARGE DATA FOR CELL C-D-8 AT 2.0A

Cell C-D-1 (0.2A) and cell C-D-8 (2.0A) were deliberately vented manually at the end of the test and subjected to IR, GC and X-ray analysis. Both cells had similar chemistries.

Gas IR analysis showed SO_2 and $SOCl_2$. GC analysis indicated that CO_2 was also present.

IR analysis of the solid salt extracted from the electrolyte revealed that the 1070 cm^{-1} peak previously observed in the IR spectra of salts extracted from the Li-limited cell (Figure 16) now appeared as a broad band centered at 1100 cm^{-1} with only a hint of the 1200 cm^{-1} peak now present as a weak shoulder. The 1070 cm^{-1} band was previously observed to shift to higher wavenumbers (1077-1085 cm^{-1}) upon overdischarging anode limited cells.[4]

An IR spectrum of the cathode showed only very weak absorptions which could only be assigned to $LiAlCl_4$. A portion of the cathode turned a deaerated NH_4OH-Naphthol yellow-S solution faint red, indicating the presence of a small quantity of $S_2O_4{}^{2-}$.

A 1.5" x 1" section of cathode was immersed in water and it bubbled vigorously. A flame held over the solution showed no reaction. The solution pH was slightly acidic. There was no apparent evidence for plated lithium.

Dry Cells at -12°C

Table 7 summarizes the performance data of the cathode limited dry cells discharged at -12°C. Cell capacities were considerably lower at this temperature, as were the pressure* increases at the end of discharge. Passivation of the anode appears to have triggered end of cell life in some cells. This is illustrated in Figure 23. Cell C-D-12 showed particularly poor capacity and was analyzed. Chemical analysis gave the same results as the cells discharged at 20°C with one exception. The solid extracted from this electrolyte no longer exhibited the IR peaks at 970 and 1330 cm^{-1}.

Wet Cells at 20°C

A summary of the performance data for the cathode limited wet cells, labelled C-W, discharged at 20°C is given in Table 8. The capacity, temperature and pressure exhibited by the cells at the end of discharge are very similar to the cathode limited dry cells. No cells exhibited hazardous behavior. Even cell C-W-16 accidentally charged for 3.57 AHr before discharge, showed no deleterious effects on subsequent discharge performance.

Cell C-W-20 was manually vented after 14 hours of overdischarge. The gas IR spectrum showed only SO_2 and a trace of $SOCl_2$. An IR spectrum of the salt extracted from the cell now showed peaks at 970 and 1330 cm^{-1}.

---

*Pressure readings at -12°C may not be accurate due to poor pressure transducer response.

TABLE 7. PERFORMANCE OF CATHODE-LIMITED DRY CELLS AT -12°C

Cell* Number	Discharge Current (A)	Current Density (mA/cm^2)	Capacity to 0.0V (A-hr)	Maximum Temp. (°C)	Maximum Pressure (psig)
C-D-10	0.2	1.0	2.88	-4	12
C-D-11	2.0	10.4	2.04	50	13
C-D-12	2.0	10.4	1.22	48	5
C-D-13	2.0	10.4	2.10	52	10
C-D-14	2.0	10.4	-	-	-
C-D-15	2.0	10.4	2.33	73	12

*Cells showed no hazardous behavior. Several cells experienced some anodic passivation which appeared to limit cell capacity. Cell C-D-14 did not discharge.

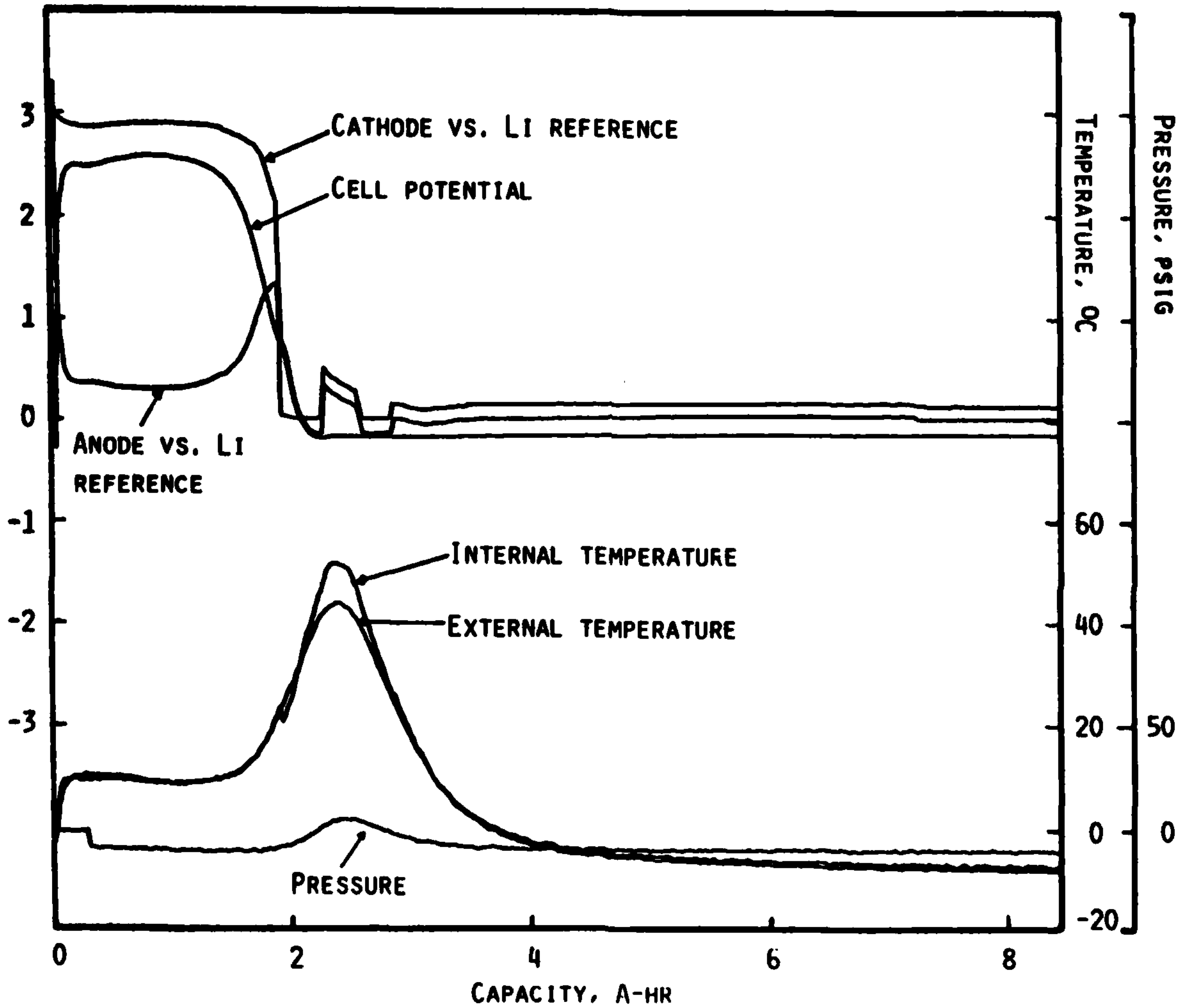

FIGURE 23. DISCHARGE DATA FOR CELL C-D-11 AT 2.0A AND −12°C

TABLE 8. PERFORMANCE OF CATHODE-LIMITED WET CELLS AT 20°C

Cell* Number	Discharge Current (A)	Current Density (mA/cm^2)	Capacity to 0.0V (A-hr)	Maximum Temp. (°C)	Maximum Pressure (psig)
C-W-16	0.2	1.0	4.65	-	25
C-W-17	0.2	1.0	4.32	32	8
C-W-18	0.2	1.0	5.24	35	10
C-W-19	0.2	1.0	5.18	34	15
C-W-20	2.0	10.4	3.41	101	142
C-W-21	2.0	10.4	3.46	117	-
C-W-22	2.0	10.4	3.78	124	151
C-W-23	2.0	10.4	3.24	115	159
C-W-24	2.0	10.4	3.23	112	190

*Cells exhibited no hazardous behavior. Cell C-W-16 was charged 3.57 A-hrs before discharge. The pressure was higher at the end of charge. The external temperature was higher in cells C-D-20 and C-D-21. Cell C-D-21 had no pressure transducer.

During the initial stage of cutting open the cell in an Argon filled dry box, the cell ignited and burst into flame blowing carbon out through the punctured hole in the bottom of the cell.

Wet Cells at -12°C

The performance data for the cathode limited wet cells are summarized in Table 9, and the data are illustrated for cells C-W-25, discharged at 0.2A and C-W-26, discharged at 2.0A in Figures 24 and 25, respectively.

Gas analysis of these two cells showed only SO_2 and $SOCl_2$.

As Figure 25 illustrates, a substantial anode polarization occurred during discharge. This was observed in other cells discharged under these conditions. The extent of polarization varied with each cell.

DISCUSSION

Although the cathodes in all cells designed as cathode-limited polarized at the end of the cells' useful life, substantial anode polarization also occasionally occurred at higher rates, especially at low temperatures. The cathode limited cells showed higher levels of heating at the end of discharge than the cells designed to be anode limited. At -12°C, the cell capacities were considerably lower, as were the pressure increases. Subtle changes occurred in the IR spectra of the extracted salts from cells discharged at 20°C and -12°C.

PARTIALLY DISCHARGED AND STORED UNCATALYZED SPIRAL CELLS

Two cells of each of the four types (A-D, A-W, C-D, C-W) of uncatalyzed spirally wound cells had been stored at ambient temperature for four months after discharging for 1.0 A-Hr at a 2.0A current. These cells were divided into two sets containing one of each type. Set one was further discharged and forced over-discharged. The Li-limited cells were tested at 2.0A and the cathode limited cells were tested at 3.0A. Discharge data are given in Table 10. No cells from set one vented. Cells from set two were opened and analyzed without further discharge.

The partially discharged cells showed very stable open circuit voltages during storage. However, there were significant changes in internal pressures. The observed pressures may be slightly off towards the end of cell storage because the pressure transducer was slowly chemically attacked by $SOCl_2$ vapor during extended storage. Plots of internal cell pressure vs. days of storage for sets one and two are shown in Figures 26 and 27, respectively.

Analysis of Forced Overdischarged Cells

The Li-limited cells were analyzed after 15 hours of forced overdischarge. No hazardous behavior was observed during forced overdischarge. IR analysis of the gases revealed SO_2 and CO_2. The gases were also collected in a liquid nitrogen cold trap. G.C. analysis of these gases confirmed that COS and $SOCl_2$ were also present.

TABLE 9. PERFORMANCE OF CATHODE-LIMITED WET CELLS AT -12°C

Cell* Number	Discharge Current (A)	Current Density (mA/cm^2)	Capacity to 0.0V (A-hr)	Maximum Temp. (°C)	Maximum Pressure (psig)
C-W-25	0.2	1.0	3.18	0	-2
C-W-26	2.0	10.4	2.12	80	17
C-W-27	2.0	10.4	1.96	54	8
C-W-28	2.0	10.4	2.38	71	13
C-W-29	2.0	10.4	1.98	78	25
C-W-30	2.0	10.4	2.06	88	32

*Cells showed no hazardous behavior. Substantial anode polarization occurred in some of these cells.

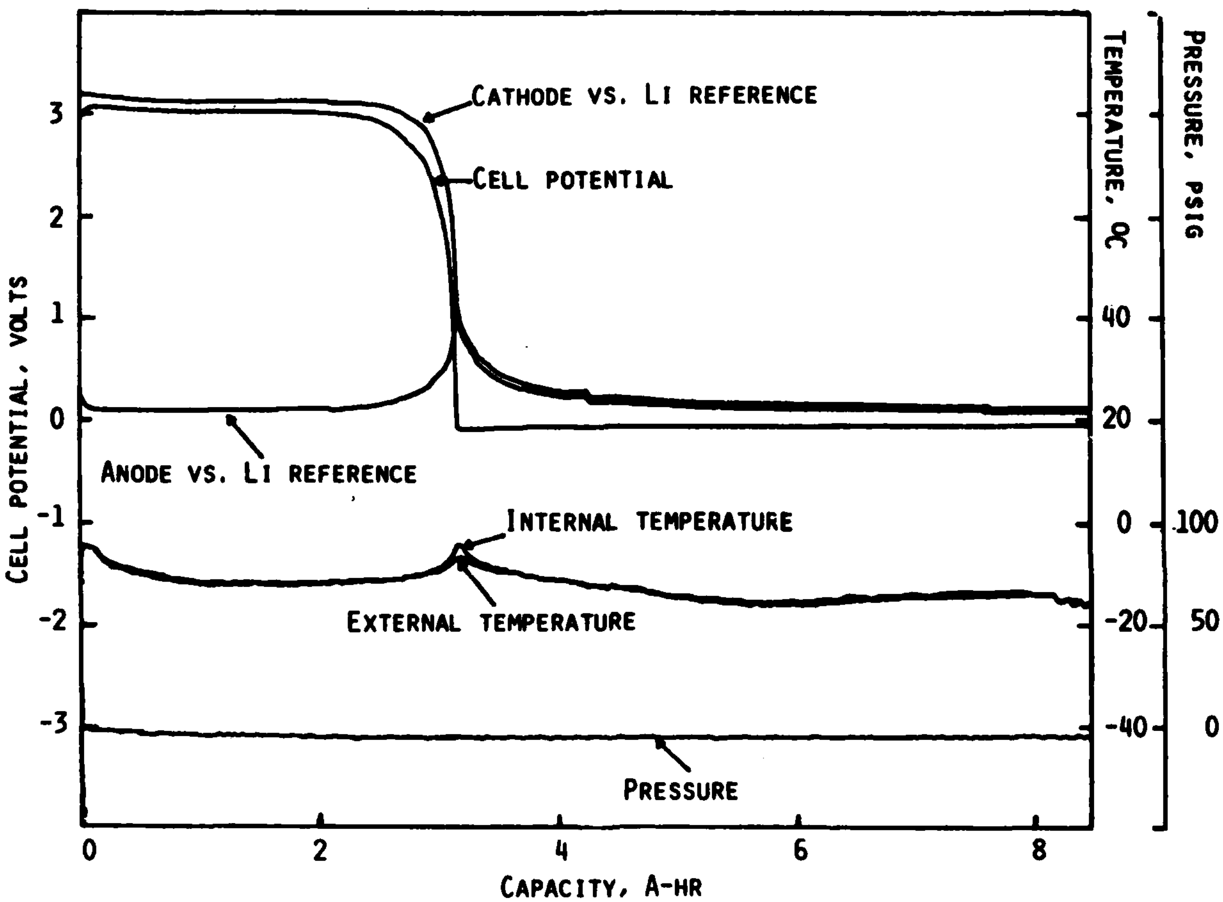

FIGURE 24. DISCHARGE DATA FOR CELL C-W-25 AT 0.2A AND −12°C

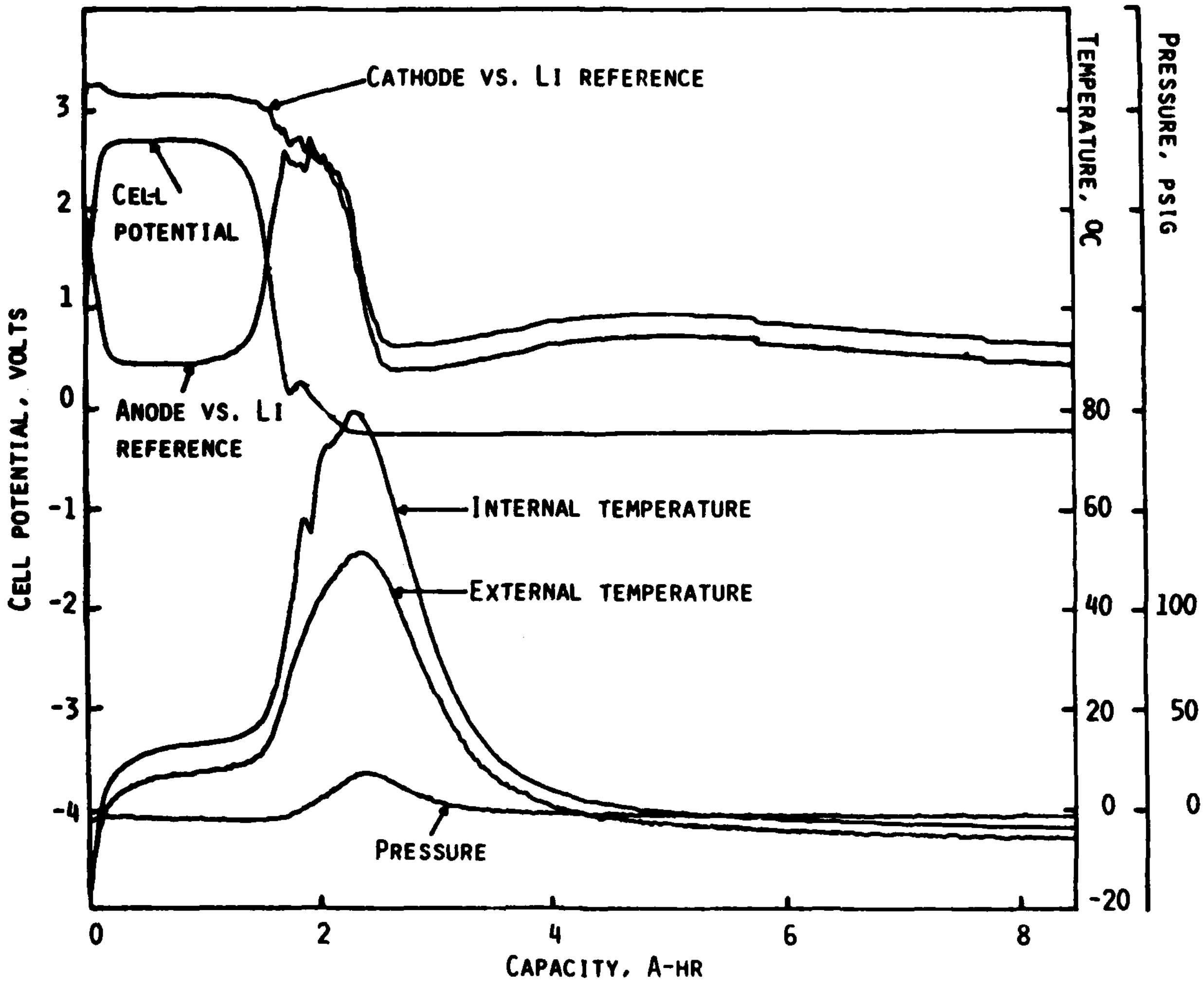

FIGURE 25. DISCHARGE DATA FOR CELL C-W-26 AT 2.0A AND −12°C

TABLE 10. PERFORMANCE OF PARTIALLY DISCHARGED AND STORED CELLS DURING FURTHER DISCHARGE AND FORCED OVERDISCHARGE

Cell Number	Discharge Current (A)	Current Density (mA/cm^2)	Capacity to 0.0V (A-hr)	Maximum Temp. (°C)	Maximum*** Pressure (psig)	Mid-discharge Voltage (V)
A-D-S-1	2.0	7.9	2.34	67*	65	2.95
A-W-S-1	2.0	7.9	2.54	60	92	3.05
C-D-S-1	2.0	10.4	3.47	133	60	
	3.0	15.6				2.50**
C-W-S-1	2.0	10.4	3.45	138	86	
	3.0	15.6				2.88**

*External temperature.
**At 3.0A rate.
***These relatively lower internal pressures seem to indicate that the calibration of the pressure transducers has changed due to corrosion during storage.

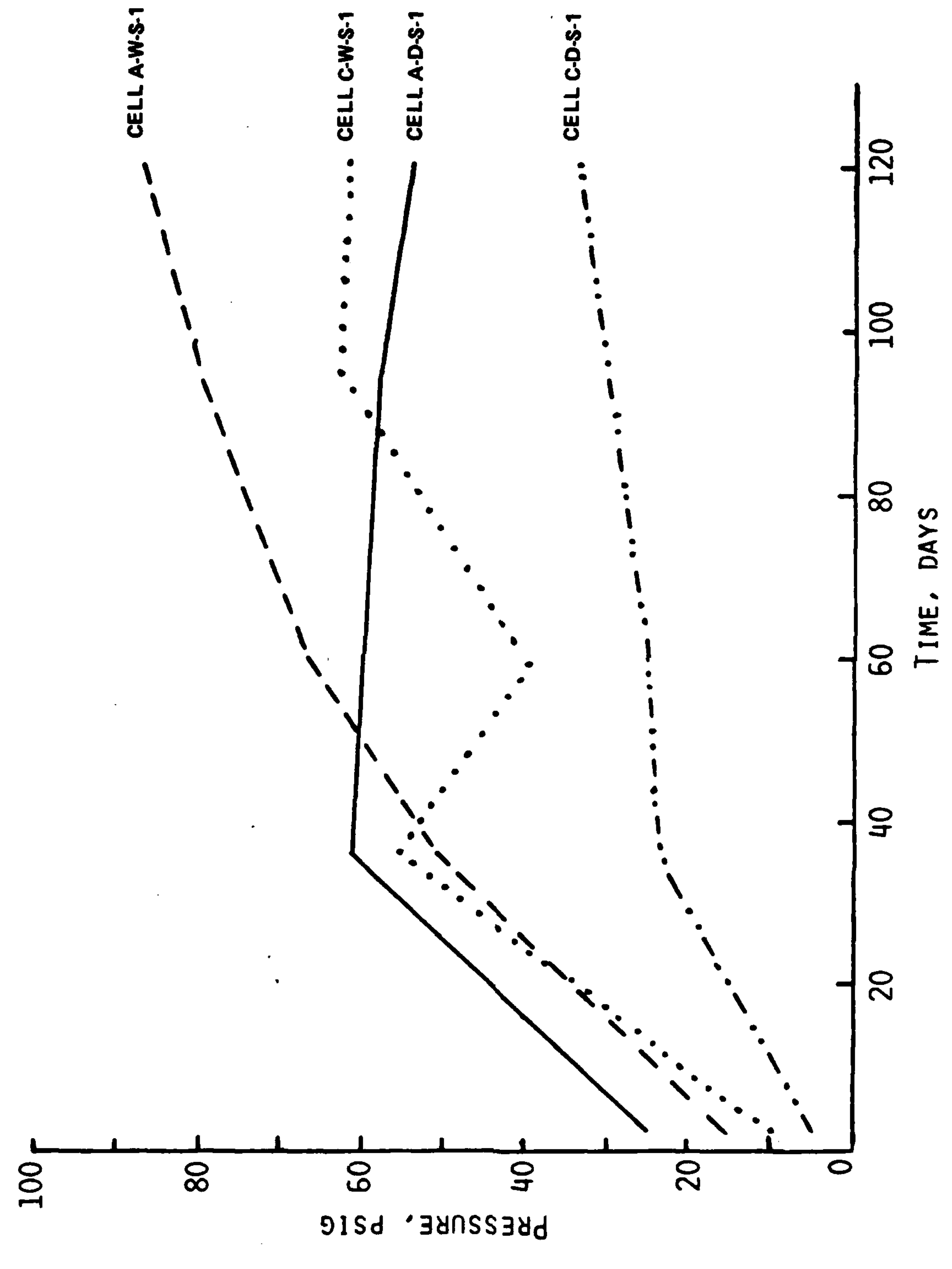

FIGURE 26. PRESSURE VS TIME CURVES FOR PARTIALLY DISCHARGED AND STORED CELLS OF SET ONE

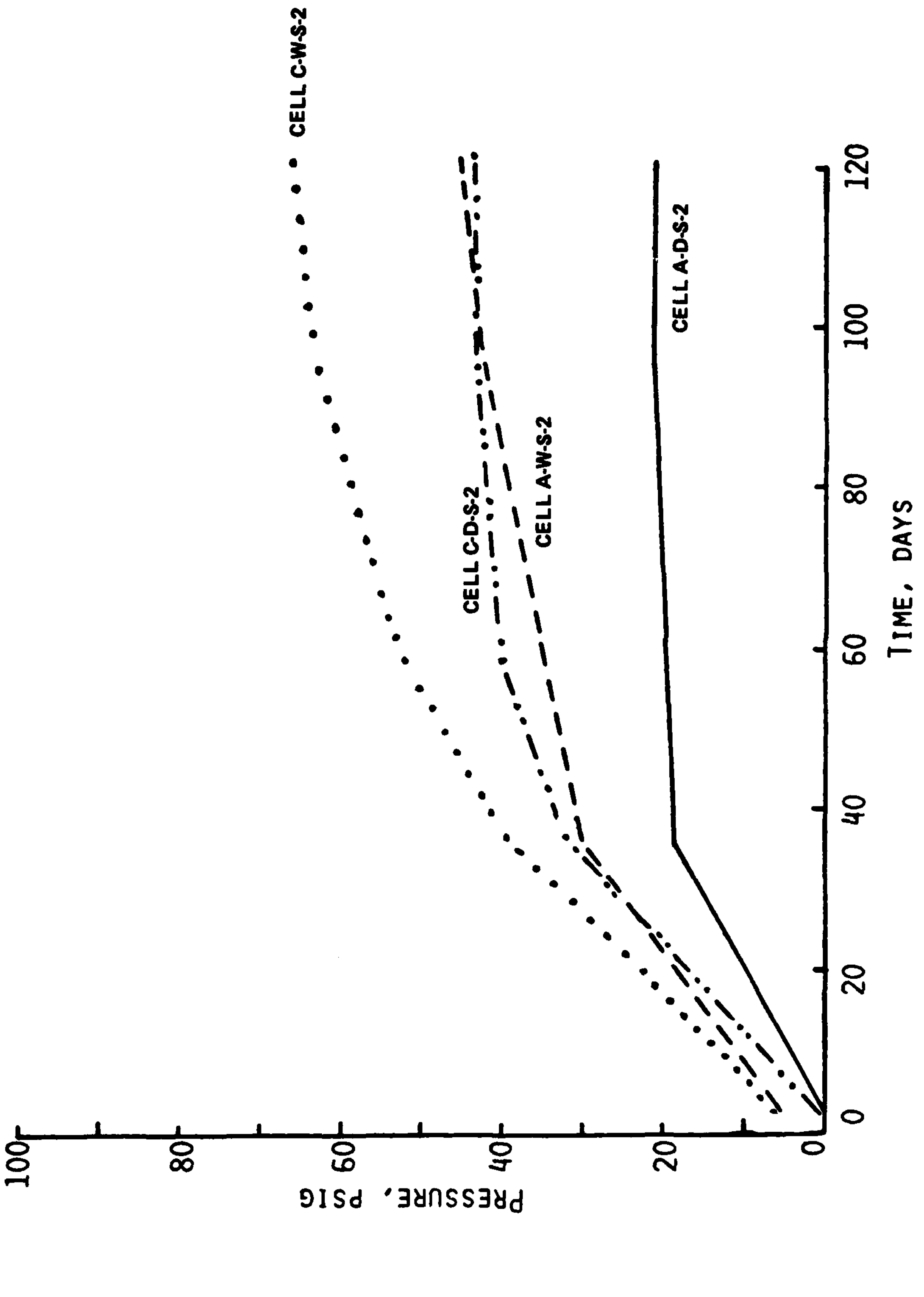

FIGURE 27. PRESSURE VS TIME CURVES FOR PARTIALLY DISCHARGED AND STORED CELLS OF SET TWO

The performance data for the wet Li-limited cell are shown in Figure 28. The IR spectra of the gases and the extracted salt are shown in Figures 29 and 30, respectively. For comparison, the performance data for the cathode limited wet cell is shown in Figure 31. Although this cell showed a much higher temperature increase at the end of discharge there was only a slight pressure increase. Again, malfunction of the pressure transducer as a result of extended storage in contact with $SOCl_2$ is indicated. Analysis of the cathode limited dry and wet cells showed no difference from that discussed earlier.

Analysis of Partially Discharged Cells

After four months of storage, chemical analyses were performed on each type (A-D, A-W, C-D, C-W) of partially discharged cells. No noticeable differences in chemistry were detected among the four cells.

The IR spectrum of the gases released from cell A-D-S-2 is shown in Figure 32. The IR spectra of both SO_2 and CO_2 possess a doublet near 2350 cm^{-1}. The SO_2 doublet is less intense with respect to its peaks at 2550 cm^{-1}. Since these two peaks are of equal intensity, this indicates a substantial amount of CO_2 has been formed in these cells during storage. G.C. analysis confirmed the presence of CO_2 and a trace of COS.

The IR spectrum of the cathode from A-D-S-2 showed no new absorptions.

The IR spectrum of the salt extracted from cell A-W-S-2 is shown in Figure 33. The spectrum is essentially the same as that obtained from the salts extracted from the overdischarged cell (Figure 30), indicating storage of partially discharged cells had little or no effect on these electrolyte soluble products.

DISCUSSION

IR analysis of extracted salts and cathodes showed the same products in both the anode or cathode limited stored cells that were either partially discharged or forced overdischarged. Analysis confirmed that reactions involving SO_2 and carbon occur during storage that produce CO_2 and some COS. Cells that vented during forced overdischarge always produced greater quantities of these gases.

HIGH RATE DISCHARGE OF SPIRALLY WOUND CELLS

Additional spirally wound cells were discharged and forced overdischarged at 3.0A at 20°C. The cathode limited cells, which showed the most likelihood of venting, were tested. The results are summarized in Table 11. None of these cells vented. A representative discharge curve of cell C-D-33 is shown in Figure 34.

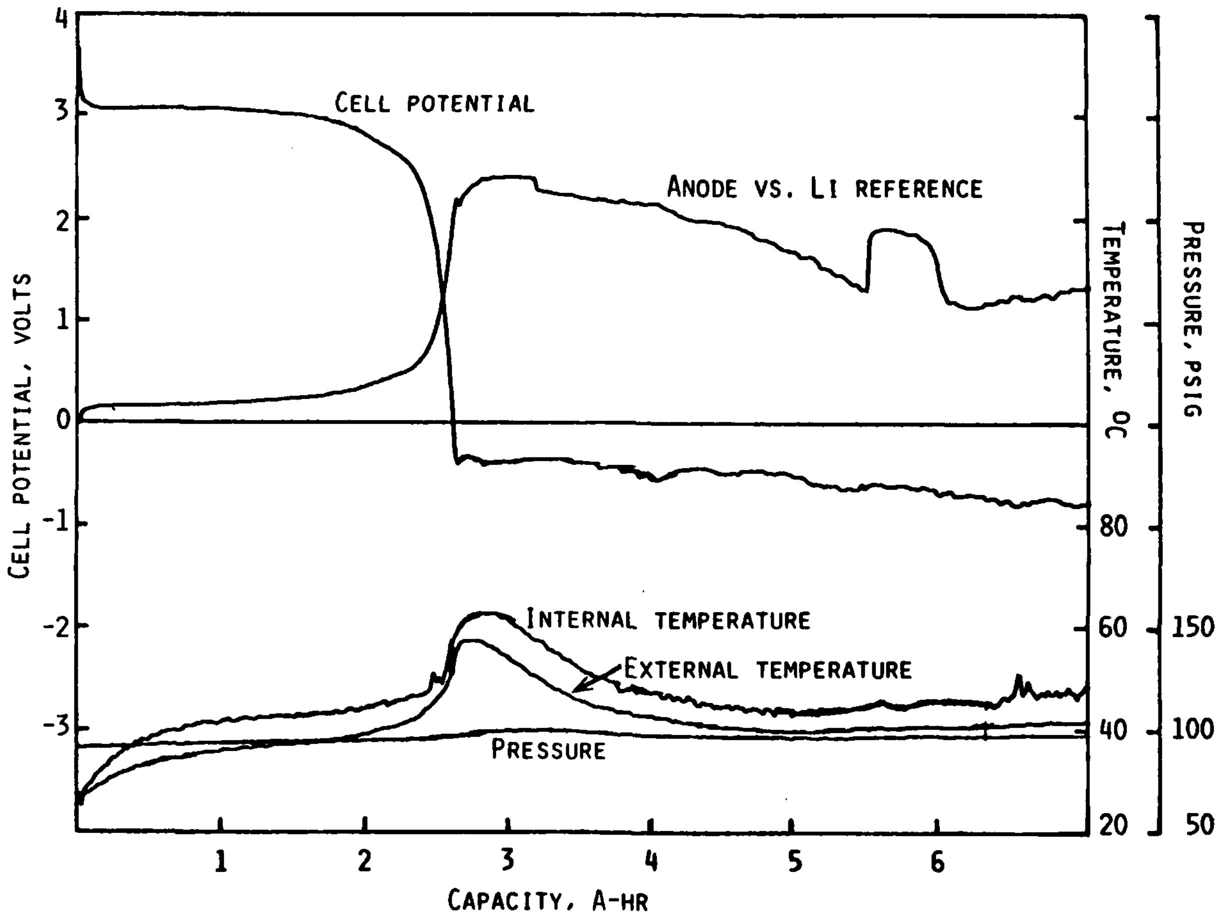

FIGURE 28. DISCHARGE DATA FOR CELL A-W-S-1 AT 2.0A

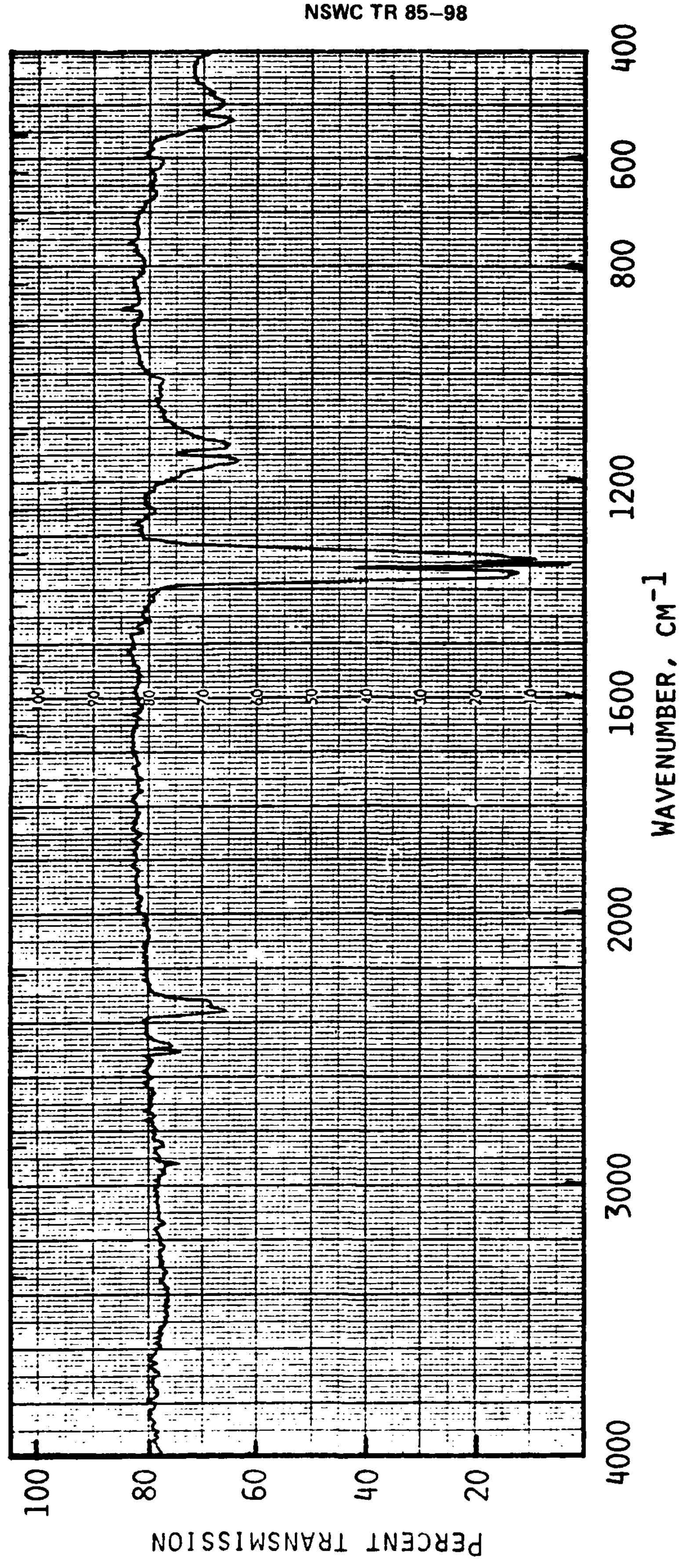

FIGURE 29. IR SPECTRUM OF GASES RELEASED FROM CELL A-W-S-1

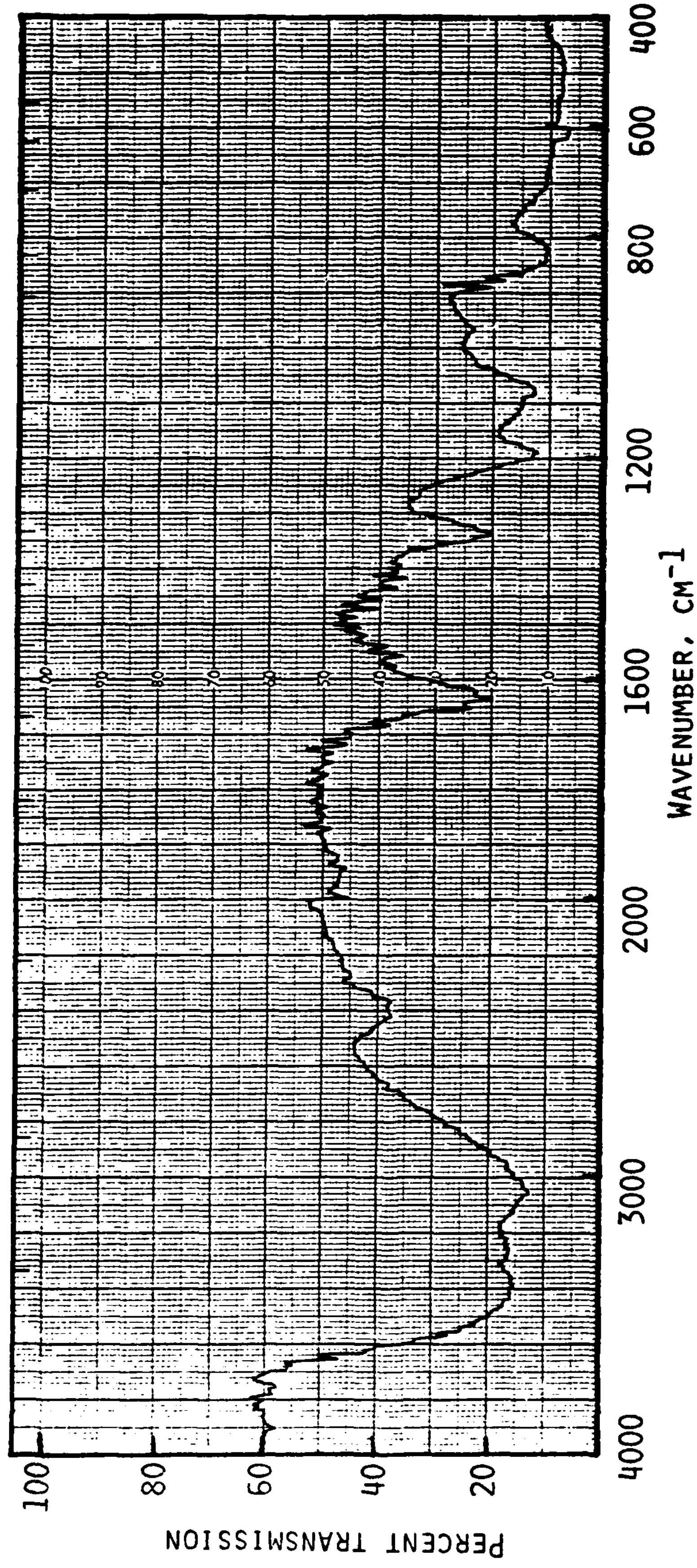

FIGURE 30. IR SPECTRUM OF SALTS EXTRACTED FROM CELL A-W-S-1

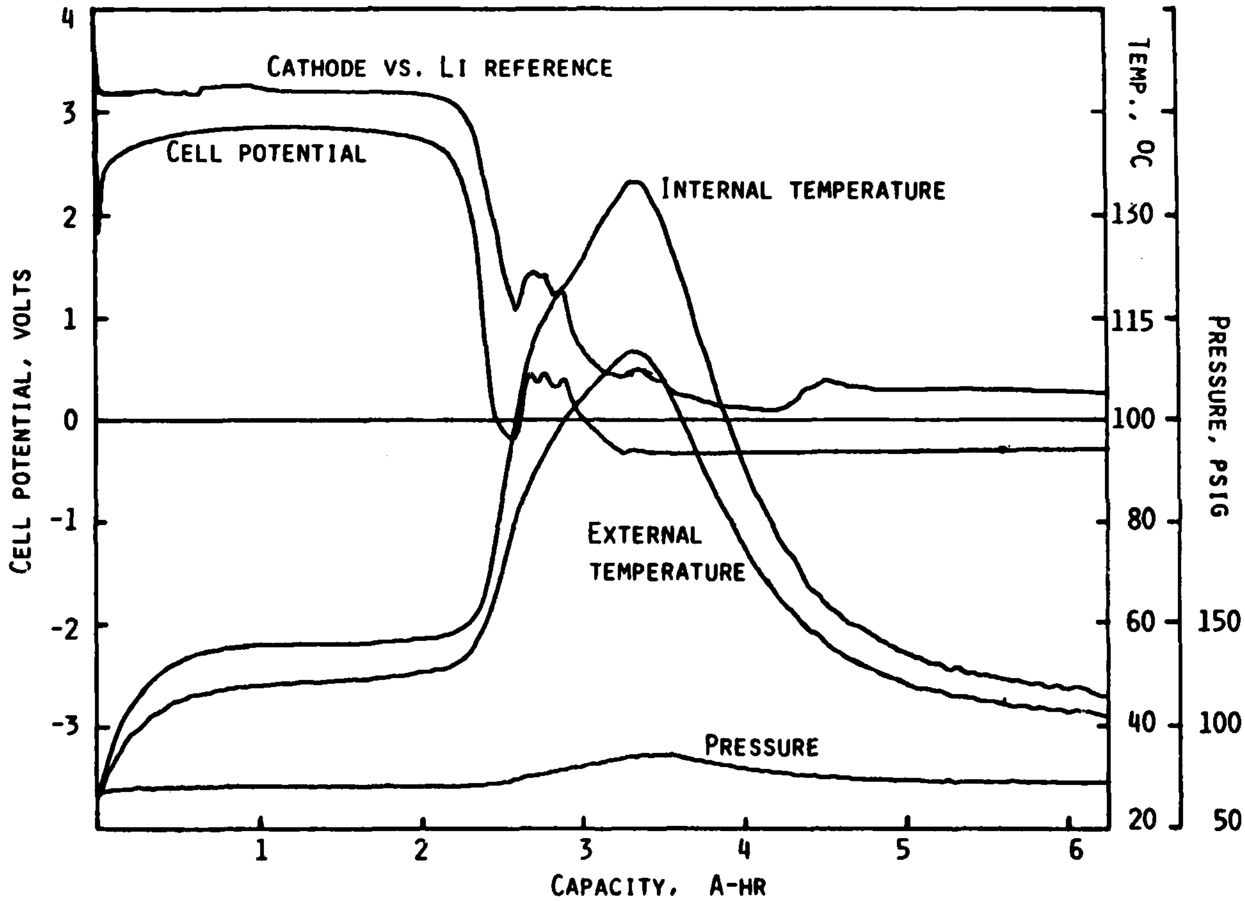

FIGURE 31. DISCHARGE DATA FOR CELL C-W-S-1 AT 3.0A

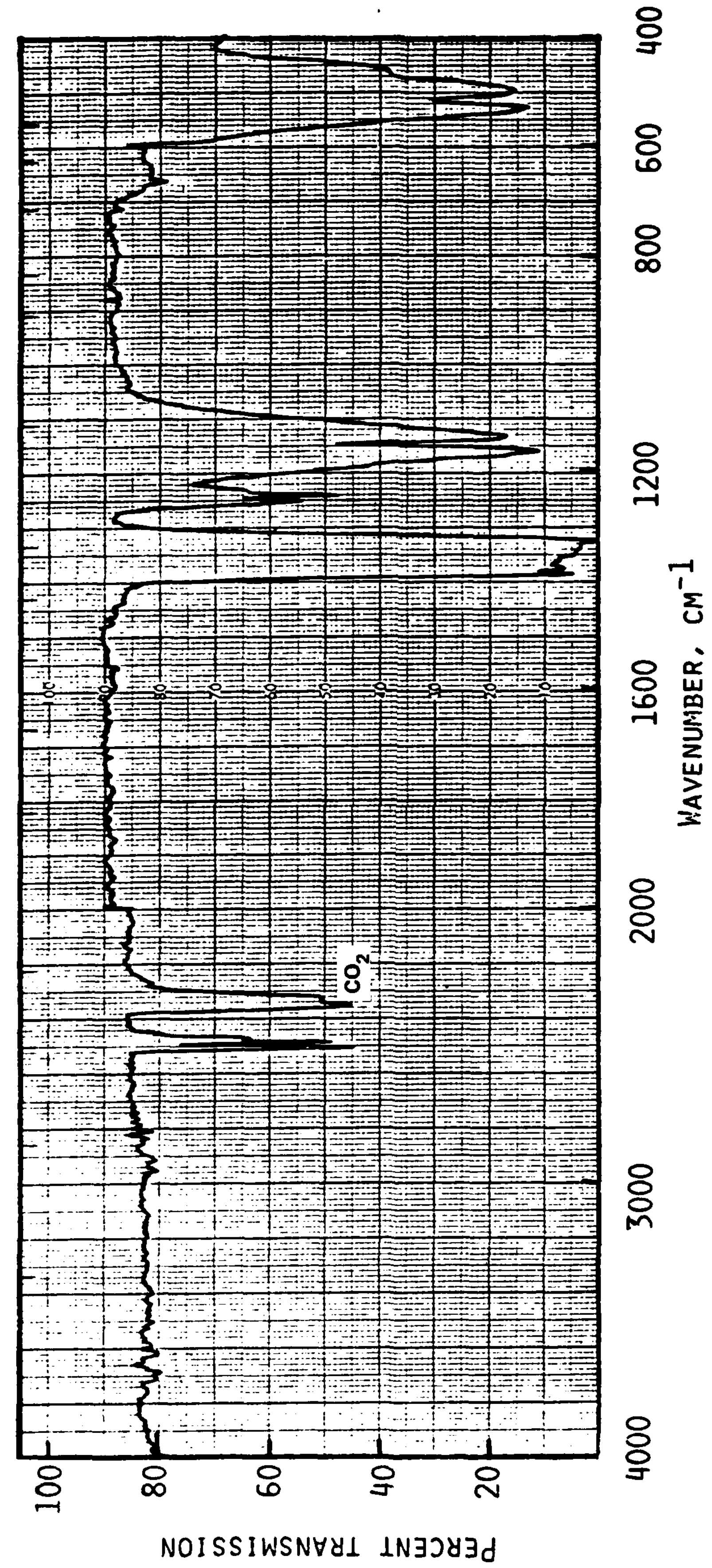

FIGURE 32. IR SPECTRUM OF GASES RELEASED FROM CELL A-D-S-2

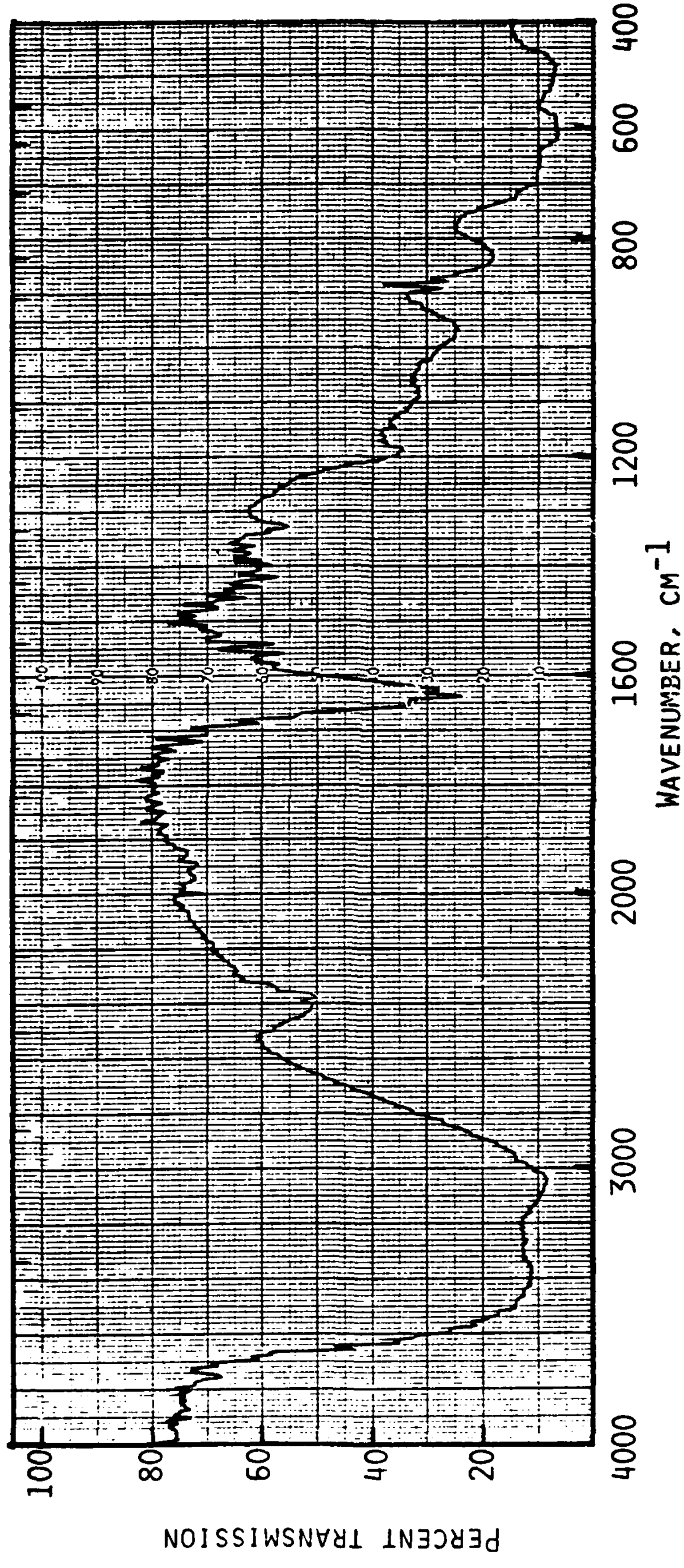

FIGURE 33. IR SPECTRUM OF SALTS EXTRACTED FROM CELL A-W-S-2

TABLE 11. HIGH RATE PERFORMANCE OF UNCATALYZED CATHODE-LIMITED DRY CELLS

Cell Number	Discharge Current (A)	Current Density (mA/cm^2)	Capacity to 0.0V (A-hr)	Maximum Temp. (°C)	Maximum Pressure (psig)	Mid-discharge Voltage (V)
C-D-31	3.0	17.2	2.63	140	151	2.80
C-D-32	3.0	17.2	2.34	114	115	2.91
C-D-33	3.0	17.2	2.39	137	195	2.88

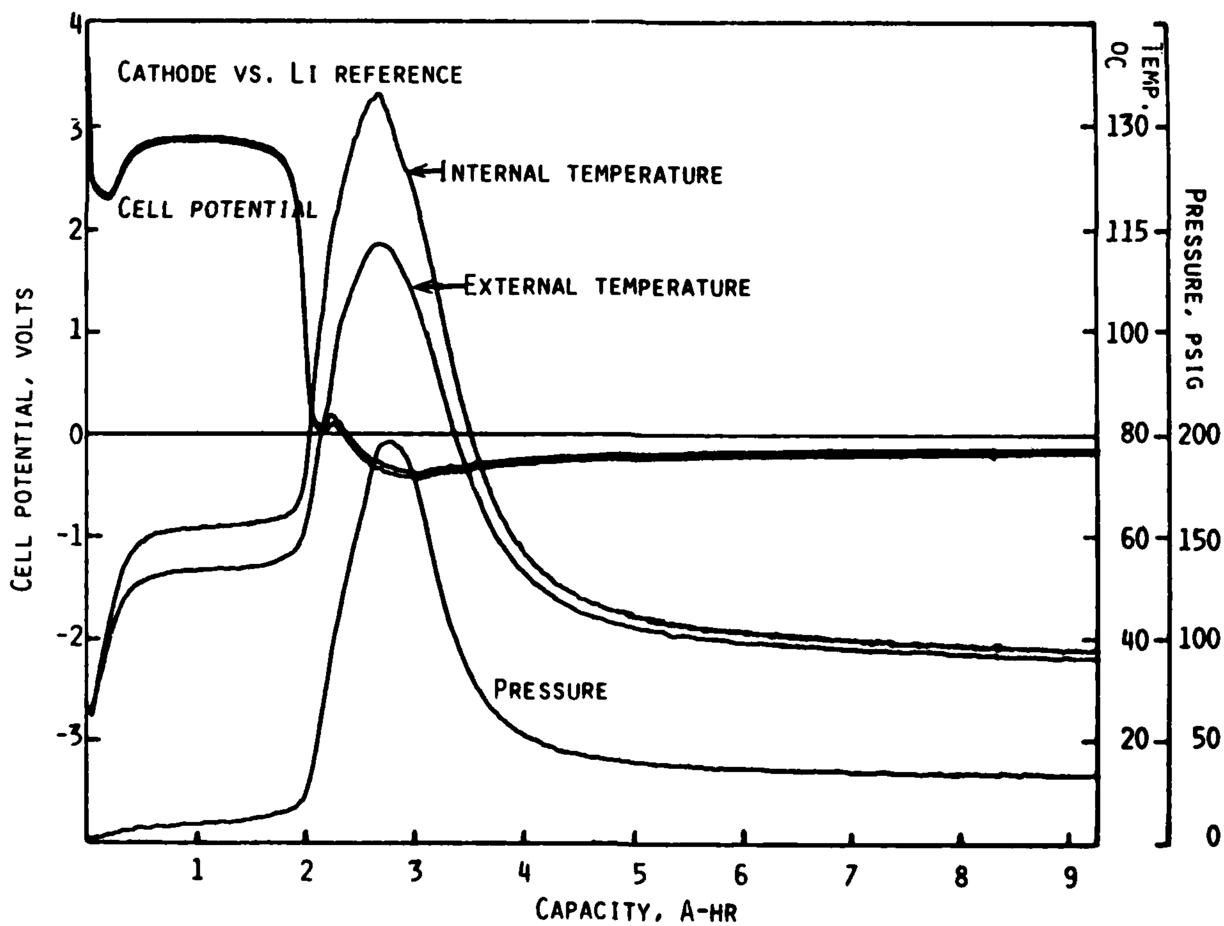

FIGURE 34. DISCHARGE DATA FOR CELL C-D-33 AT 3.0A

DISK ELECTRODE CELLS

The component weights and surface areas of the disk-type cells were maintained as closely as possible to those of the spirally wound cells in order to better compare the performance data. In doing this, the selection of the best cell design and safety were compromised. Of the 24 cells built, only 8 were tested before it became apparent that the selected cell design led to potential safety hazards. One of the cells vented violently and the additional tests were discontinued (see Section G).

Eight uncatalyzed disk cells were tested. These are labelled; D-A-D, Disk-Li-limited-Dry; D-A-W, Disk-Li-limited-Wet; D-C-D, Disk-Cathode-limited-Dry; and D-C-W, Disk-Cathode-limited-Wet.

The cells were discharged at 20°C. In several cells the internal thermocouple failed. Both of the D-C-W cells discharged at 3.0 and 4.0 amp rates vented during forced overdischarge without an incidence of explosion. None of the anode limited cells vented during forced overdischarge, although the pressure in cell D-A-W-4 reached 328 psig. The data are summarized in Table 12. The discharge data for a cathode limited wet cell discharged at the 4.0A rate are shown in Figure 35.

REPORT ON A HAZARDOUS EVENT WITH A DISK CELL

Two cathode limited, uncatalyzed, dry electrolyte disk cells were tested. Cell D-C-D-7 showed the proper open circuit voltage but when a 3.0A current was applied, the voltage immediately dropped to a negative value. The cell could not be discharged at 2.0A. At 1.0A, the voltage was -0.2V. The cell was subsequently left on open circuit, housed still inside the test chamber.

Cell D-C-D-8 showed the same behavior. Despite the proper open circuit voltage, when 4.0A was applied to the cell, the voltage fell to negative values, and at 2.0A the potential was -8.0V.

The two cells, still housed in safety test chambers, designed to vent at 50 psig, were taken away from the test station and set aside on the laboratory bench. About 4.5 to 5.0 hours later, cell D-C-D-8 violently exploded venting the safety chamber and filling the laboratory with smoke. A visual inspection showed the cell had split open along the entire length of the wall of the can. The vent at the bottom was opened but evidently the explosion was too rapid for it to function properly.

SUMMARY

In an attempt to match the specifications of the spiral cells, the C size disk electrode cells required very many layers of electrodes. It is suspected that this choice of cell design may have led to an internal short on some of the hand assembled cells.

TABLE 12. PERFORMANCE OF UNCATALYZED DISK ELECTRODE CELLS

Cell Number	Discharge Current (A)	Capacity to 0.0V (A-hr)	Maximum Internal Temp. (°C)	Maximum External Temp. (°C)	Maximum Pressure (psig)	Mid-discharge Voltage (V)
D-A-D-1	4.0	2.35	-	110	195	2.80
D-A-W-2	4.0	2.15	126	121	170	2.80
D-A-D-3	3.0	2.86	155	152	175	2.75
D-A-W-4	3.0	1.74	-	155	328	2.75
D-C-W-5	4.0	2.78	125	121	200	2.90
D-C-W-6	3.0	1.59	-	157	155	2.73

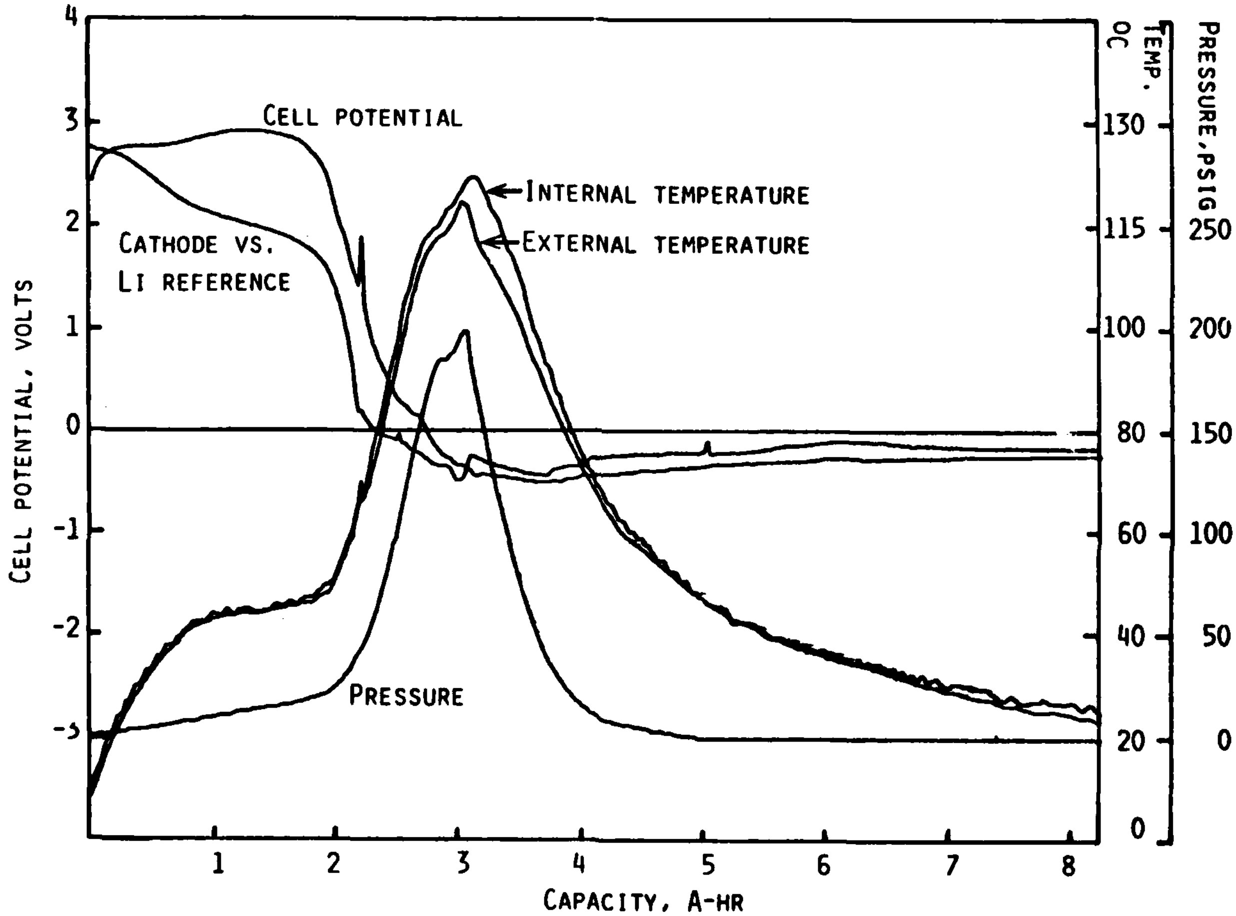

FIGURE 35. DISCHARGE DATA FOR CELL D-C-W-5 AT 4.0A

CHAPTER 4

SUMMARY AND CONCLUSIONS

Pressure-temperature measurements of spirally wound high rate Li/$SOCl_2$ cells indicated that the actual pressure generated in the cell is much lower than that calculated on the basis of the discharge reaction,

$$4Li + 2SOCl_2 \rightarrow 4LiCl + SO_2 + S$$

The lower pressure may be due to one or more of the following effects: (i) complexation of the SO_2 by $LiAlCl_4$,[5] (ii) absorption of SO_2 by carbon,[6] and (iii) the presence of a significant amount of low volatile $(SO)_x$ rather than its decomposition products S and SO_2.[7] Further studies are required to ascertain the exact mechanism by which the cells maintain lower internal pressures. It should be noted, however, that were it not for a built-in SO_2 scavenging mechanism, the Li/$SOCl_2$ cell would produce excessive internal pressures capable of potential violent venting, especially during early stages of cell reversal.

The individual electrode potential data indicate that a substantial fraction of the internal heating at the end of cell life is associated with cathode polarization. Since Li-limited cells show little or no cathode polarization towards the end of cell discharge, the temperature and pressure maxima reached in the Li-limited cell were considerably lower than that in the cathode-limited cell. The pressure-temperature data obtained from constant current discharges are more relevant to series connected batteries than single cells. During a constant load discharge, the current at the end of cell-life when the cell voltage approaches zero volt would be substantially reduced. Consequently, the associated internal heating would be significantly less compared to that in a constant current discharge.

It was possible to force overdischarge cathode limited cells for extended periods of time with charge inputs exceeding the amount of Li originally present in the cell. This suggests cell short circuiting accompanying plating of Li onto the cathode. The short-circuited cell apparently shunts current as if it were a resistor. The fact that substantial amounts of Li remain on the anode of an extensively overdischarged cell supports this hypothesis. This situation did not seem to pose a special safety problem.

None of the Li-limited cells exhibited safety hazards during forced overdischarge. The Li utilization in the 0.2 to 2.0A current range was virtually 100 percent in the Li-limited cells. Anode polarizations caused by poor electrical contact between Li and the current collector grid, as found previously [3], were not observed, apparently explaining the lack of safety hazards in this study.

Anodic polarization was observed at the end-of-life of many cathode-limited cells. This phenomenon appeared more pronounced at high currents and low temperatures and is not well understood. It may be due to a non-homogeneous current distribution towards the end of discharge and/or a concentration polarization arising from crystals of LiCl and/or $LiAlCl_4$ forming on the Li surface.

Li-limited cells also showed an ability to withstand forced overdischarge for extended periods of time. This capability may be due to regenerative cell processes.[8]

All of the cathode-limited cells also exhibited safe behavior in the 0.2 to 2.0A range of discharge and overdischarge. Even at 3.0A tests most cells exhibited safe behavior. However, one cell tested at 3.0A vented during forced overdischarge. The venting, accompanied by a sudden increase in temperature, occurred without any prior warnings of pressure, temperature or voltage changes.

The self-discharge in 30 percent discharged cells stored for four months at ambient temperature was virtually undetectable. There was also little new chemistry identifiable as due to the storage.

The present data appear to corroborate the idea that a safe cell design would be Li-limited with provisions for a complete utilization of Li at the end of discharge over a wide range of current densities encompassing the use loads of the battery at various temperature environments. The catalyzed cells discussed in the second report in this series seem to provide these advantages.

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